

Separation Schemes – Tips and Notes

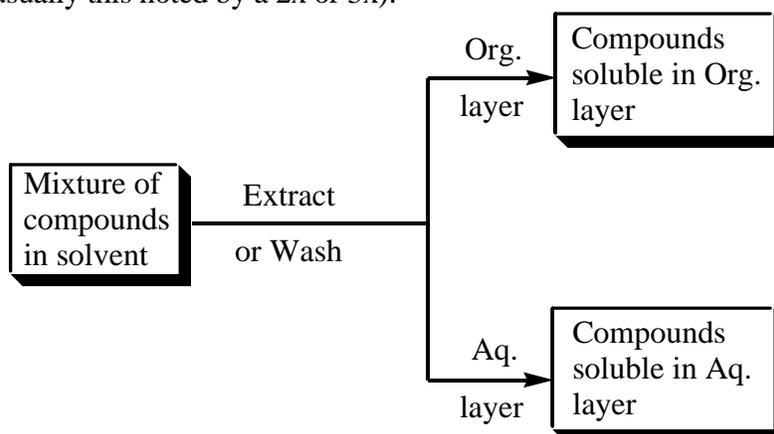
A **Separation Scheme** tells you (or your lab partner) where your product and any by-products are at any point during the isolation of your final product from a chemical reaction. Whenever there is the physical separation of two immiscible layers, a solid from a solid/liquid mixture or the removal of a solvent from the final product, the **Separation Scheme** details where each product or by-product is (or should be). It is a useful guide to you or anyone else performing the same experiment that shows how to get the final product in pure form, devoid of any of the by-products or solvents.

The start of any **Separation Scheme** is typically the end of the reaction, when all of the ingredients are still in the reaction flask, but the reaction is considered complete. Typical physical manipulations of the reaction mixture at the end of the reaction include, extracting, washing, rinsing, filtration, evaporation of solvent, and drying.

Extracting or Washing. This involves mixing immiscible solvents, usually using a separatory funnel – one layer will be on top and the other will be on bottom. In terms of the actual procedure for using a separatory funnel, extracting and washing are identical. Organic compounds, inorganic compounds, or ionic forms of organic compounds (e.g., ammonium ions or carboxylates) will partition between the two solvents and will “choose” a solvent in which it has the greater solubility. This will either be the aqueous (aq.) layer (H_2O) or the organic (org.) layer. Separation of these layers using the separatory funnel constitutes a “separation” in a **Separation Scheme**. Examples of the most common aqueous solutions and organic solvents are shown below:

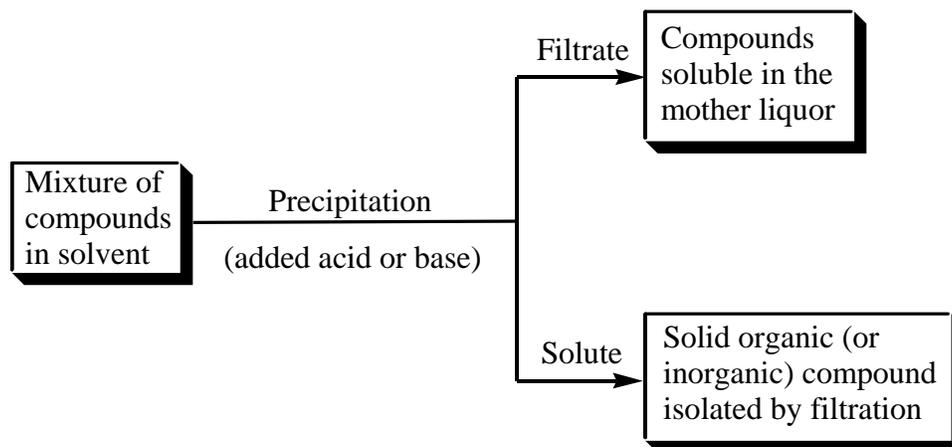
Aqueous	Organic Solvent (in order of polarity)
water	ethyl acetate (EtOAc)
HCl solution (acidic)	diethyl ether (Et_2O)
NaOH solution (basic)	dichloromethane (aka methylene chloride, CH_2Cl_2)
NaHCO_3 solution (mildly basic, usually saturated)	hexanes
NaCl solution (usually saturated)	petroleum ether (pet. ether)

Since all of the aqueous and organic combinations listed above are immiscible, when mixed in a separatory funnel there will always be a “top layer” and a “bottom layer”. Which layer is on top? The most dense solvent is on the bottom. Of the organic solvents, only dichloromethane (CH_2Cl_2) is more dense than water (however on rare occasion some aqueous solutions with high concentrations of dissolved materials can be more dense than dichloromethane). All other solvents are less dense and will be on top. Frequently, mixtures are extracted or washed more than once (usually this noted by a 2x or 3x).



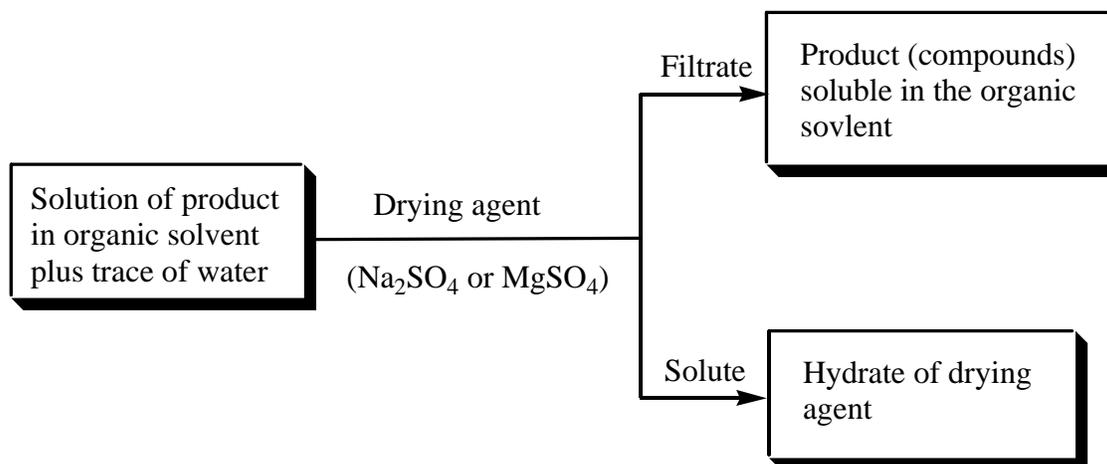
Precipitation. If a heterogeneous solid/liquid mixture is produced (such as with acidification or basification) and is separated, then the two components after separation are known as the *filtrate* (liquid) and the *solute* (solid), whether by *gravity* or *vacuum filtration*. In either case, when the mixture is separated, it would be noted as shown below.

(Note that *filtrate* as a noun refers to the liquid produced after a *filtration*, though as a verb describes the process of filtering and is in practice used very infrequently.)

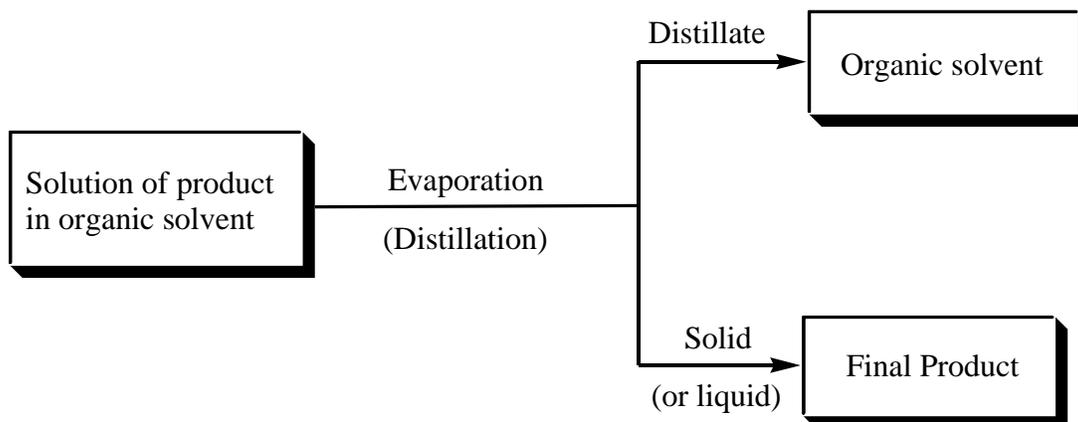


Drying agents. When a solution of an organic compound is dried using a *drying agent* (such as anhydrous Na_2SO_4 or MgSO_4), the resulting mixture is similar to that from a Precipitation process – the resulting mixture is an organic compound in solution and a solid (the hydrate) that requires removal by filtration. In this case the solid is not the desired material, but rather the filtrate, in which the compound of interest is still in solution. In this case, the filtrate is usually obtained by either *decanting* or by *gravity filtration* and the solute (hydrates, $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ or $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) is discarded.

(Note that the drying agent never “dissolves” in an organic solvent, but simply goes from anhydrous to hydrated)



Evaporation. Usually, when the final product is in a solution with an organic solvent, such as after extraction and the use of a drying agent, the solvent is removed by evaporation. This process is officially *distillation*. Common methods of evaporation include using a *rotary evaporator* (RotoVap) or a hot plate in a hood. Both methods accomplish the same end: the result is a separation of the solvent and the solid or liquid product through *distillation*. The solvent vapor might be condensed and recovered (RotoVap) or might not (hot plate in the hood).



Crystallization or Distillation. Although both techniques are considered vitally important purification methods for organic solids and liquids, they are not usually included in a separation scheme.

Putting it all together. A complete separation scheme includes all of the above items in the order of when they are performed. Follow the product of interest (the final product)! Below is a schematic form of a **Separation Scheme**, which leads to the purified final product. Note that each step of the separation requires two arrows. Use this as a guide to produce your own scheme.

