DETERMINATION OF SOLUBILITY CLASS

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Solubility class determination gives an idea about the type of the functional group present in the compound, the polarity and molecular weight of the compound, and the nature of the compound (acidic, basic, neutral).This is accomplished by testing the solubility of the compound in either of the following sets of solvents:

distilled water, 5% sodium hydroxide solution, 5% sodium bicarbonate solution, 5% hydrochloric acid solution, and cold concentrated sulfuric acid, or distilled water and ether. It is well known that hydrocarbons are insoluble in water because of their non polar nature. If an unknown compound is partially soluble in water, then this indicates that a polar functional group is present. Additionally, solubility in certain solvents often leads to more specific information about the functional group.

For example:

benzoic acid is insoluble in water, but is converted by 5% sodium hydroxide solution to a salt, sodium benzoate, which is readily water soluble. In this case, then, the solubility in 5% sodium hydroxide solution of a water insoluble unknown is a strong indication of an acidic functional group. Prediction of the molecular weight and size may sometimes be obtained from the result of solubility tests.

For example:

in many homologous series of mono functional compounds, the members with fewer than about five carbon atoms are water soluble, whereas the higher homologs are insoluble .

The first step to follow is to test the solubility of the compound in water.

Generally and for solubility classification purposes, the compound is said to be soluble in any solvent if it dissolves to the extent of about 3 % (0.1 gm/3 ml or 0.2 mL/3 mL). This is achieved by dissolving about 0.1 gm of the solid compound or 3-4 drops of the liquid compound in gradually increasing volumes of the solvent up to 3 ml (max. allowed volume is 3 ml) with shaking

Classification to determine whether the compound is soluble or insoluble in that solvent. When solubility in dilute acid or dilute base is being considered, the significant observation to be made is whether it is significantly more soluble in aqueous acid or aqueous base than in water. Such increased solubility is the desired positive test for acidic or basic functional groups. Below is a very useful scheme for solubility classification

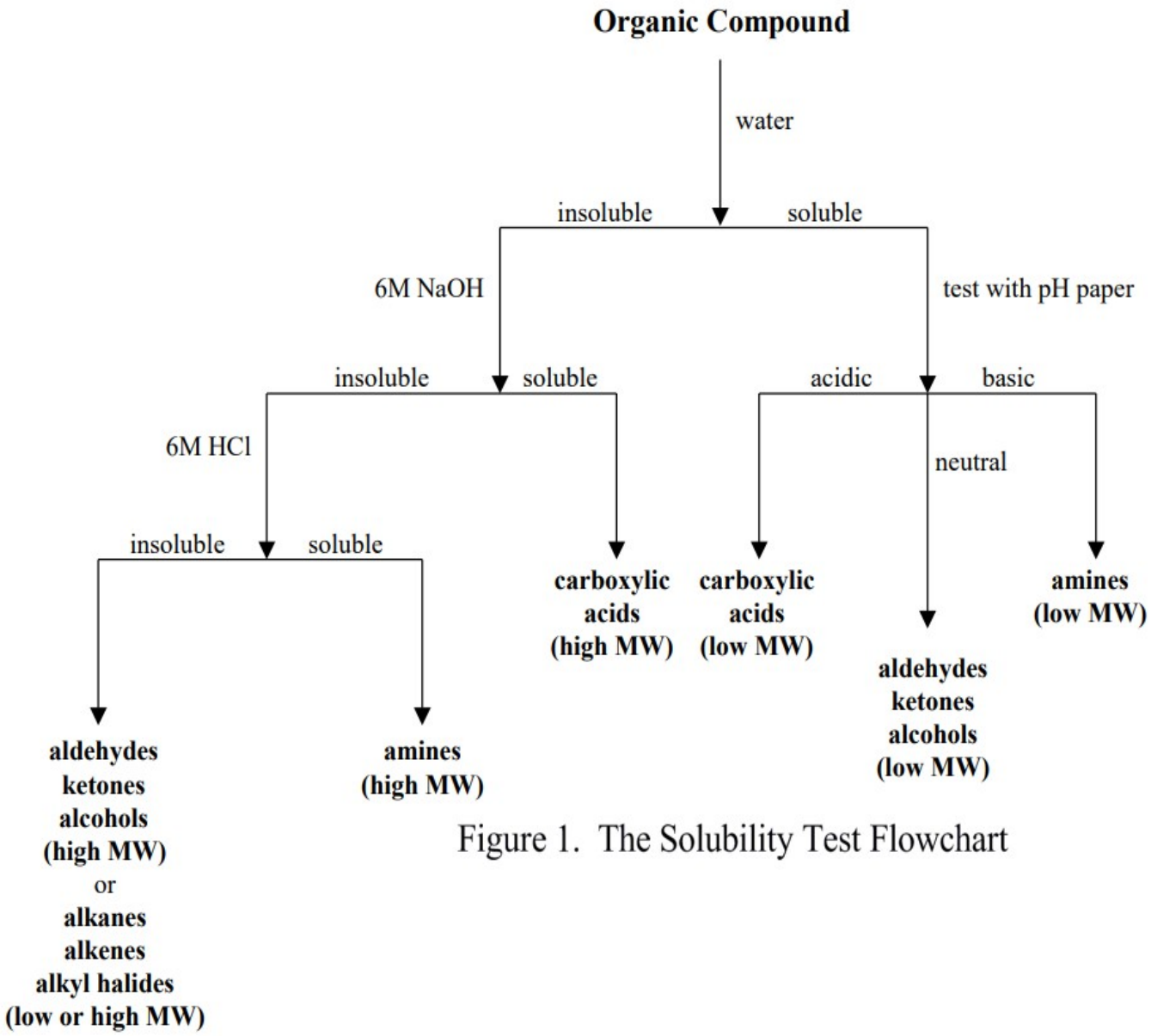

Figure 1. The Solubility Test Flowchart

WATER AS SOLVENT

Water is a polar solvent with a dielectric constant equals to 80. It has the ability to form hydrogen bonding and can act either as an acid or a base. Therefore it can dissolve.

-Salts of ammonium ion (RNH4) or organic acids salts with

-alkali metal cations (RCOO).

-Ionic compounds.

- Polar compounds “like dissolves like”.

- Organic compounds with low molecular weight (less than 5 carbon atoms) such as alcohols, aldehydes, ketones, and carboxylic acids.

Water is useful to determine the degree of acidity of a compound, even if the compound is insoluble in water, using litmus paper (acidic, basic, neutral). Water is the first solvent used to determine the solubility class of a compound. If the compound is water soluble, the next step is to test its solubility in ether.

ETHER AS SOLVENT

Ether is a non-polar solvent having a dielectric constant of 4.3. It cannot form hydrogen bonding (unassociated liquid). Therefore, it differs from water in that it cannot dissolve ionic compounds such as salts. It dissolves most water insoluble compounds; therefore, in the determination of solubility class, the importance of ether is for water soluble compounds only and no further solubility tests using the remaining solvents are to be done.

Accordingly two probabilities are there:

1. Compounds soluble in both water and ether, and these compounds:

- are non-ionic.

- contain five or less carbon atoms.

- contain an active group that is polar and can form hydrogen bonding.

- contain only one strong polar group.

This division of compounds is given S1class and includes, e.g., aldehydes, ketenes, and aliphatic acids.

2. Compounds soluble in water only (but not in ether), these compounds:

-are ionic.

- Contain two or more polar groups with no more than four carbon atoms per each polar group. This group is classified as S2 class and includes ionic salts such as salts of carboxylic acids and amines and compounds with more than one active group such as poly hydroxylated compounds and carbohydrates. Note that solubility in ether is tested only for water-soluble compounds. For water insoluble compounds use the left side of the solubility classification scheme, i.e. test solubility in sodium hydroxide rather than ether

5% NAOH & 5% NAHCO3

Water insoluble compounds must be tested first in 5% sodium hydroxide solutions which is a basic solvent. It reacts with water insoluble compounds that are capable of donating protons such as strong and weak acids. The stronger the acid, the weaker the base it can react with. Water insoluble compounds that dissolve in 5% sodium hydroxide solution must also be tested for solubility in 5% sodium bicarbonate solution. Therefore, for water insoluble acidic compounds sodium hydroxide solution is considered as a detecting solvent whereas sodium bicarbonate solution is called as a sub classifying solvent since it can react with strong acids only. That is, these two solvents give an idea about the acidity degree of the compound. Note that testing solubility in 5% sodium bicarbonate solution is not needed if the compound is insoluble in 5% sodium hydroxide solution, but rather, 5% hydrochloric acid solution should be used.

Two probabilities are there:

1. Compounds soluble in both bases.

This group is given class A1. This class includes strong acids that have the ability to react with weak bases (carboxylic acids) and phenols with electron withdrawing groups (e.g.,–NO2). Protons are weakly attached and can be given easily.

2. Compounds soluble in 5% sodium hydroxide solution only.

This group is given class A2 and it includes phenols, amides, and amino acids (weak acids). sodium hydroxide solution, but rather, 5% hydrochloric acid solution should be used

HCL 5%

If the compound is insoluble in water and sodium hydroxide solution (and, hence, insoluble in sodium bicarbonate solution too), this means that the compound is not an acid but, rather, is either a basic compound or a neutral compound. 5% hydrochloric acid solution, which can dissolve basic compounds such as amines (RNH2), is used for such a compound. If the compound is soluble in this solvent, then it is given class B. This class includes primary, secondary, and tertiary amines.

COLD CONCENTRATED H_2SO_4

If the compound is insoluble in water, 5% sodium hydroxide solution, and 5% hydrochloric acid solution, solubility in cold concentrated sulfuric acid should be tested. If the compound is soluble in this acid, it belongs to class N which includes neutral compounds such as high molecular weight alcohols, aldehydes, ketones, esters, and ethers (more than four carbon atoms), and unsaturated hydrocarbons. On the other hand, compounds that are insoluble in cold concentrated sulfuric acid belong to class I which includes inert aliphatic (saturated) hydrocarbons, aromatic hydrocarbons, haloalkanes, and aryl halides.