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A REVIEW ON FUNDAMENTALS OF WET CHEISTRY

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ABSTRACT

Wet Chemistry involves the two branches viz, **Pharmaceutical Chemistry** and **Pharmaceutical Analysis** with various instrumental and non-instrumental techniques which are followed or practiced in this filed with various methods like identification tests, synthesis, separation, distillation, titration, preparation of dilute liquids, etc. This review deals with discussion, performing, evaluating some Chemistry and Analytical branch related techniques and some experiments performed in Laboratories as well as our day to day life .In this following review the certain topics like Wet Chemistry involved in volumetric analysis ,chromatography are included in the Instrumental techniques where as wet chemistry involved in separation, extraction, synthesis are included in the non-instrumental techniques with the suitable examples.

Keywords: Wet Chemistry, Volumetric Analysis, Spectral Analysis, Chromatography, Liquids Separation, Non-instrumental techniques, New Product.

INTRODUCTION[1]

Wet Chemistry is the branch of analytical chemistry which deals with the studies involved like titration, separation, synthesis, identification tests, etc. Wet chemistry includes activities like mixing, measuring, weighing of liquids. Also wet chemistry is involved in the determination of viscosity, density, pH, temperature, vapor pressure, surface tension.Wet chemistry also called as bench chemistry, the reason behind this is; in the wet chemistry the experiments are performed only with liquids, using liquids on the laboratory benches.In our daily life too, we undergo some activities involved in the wet chemistry.

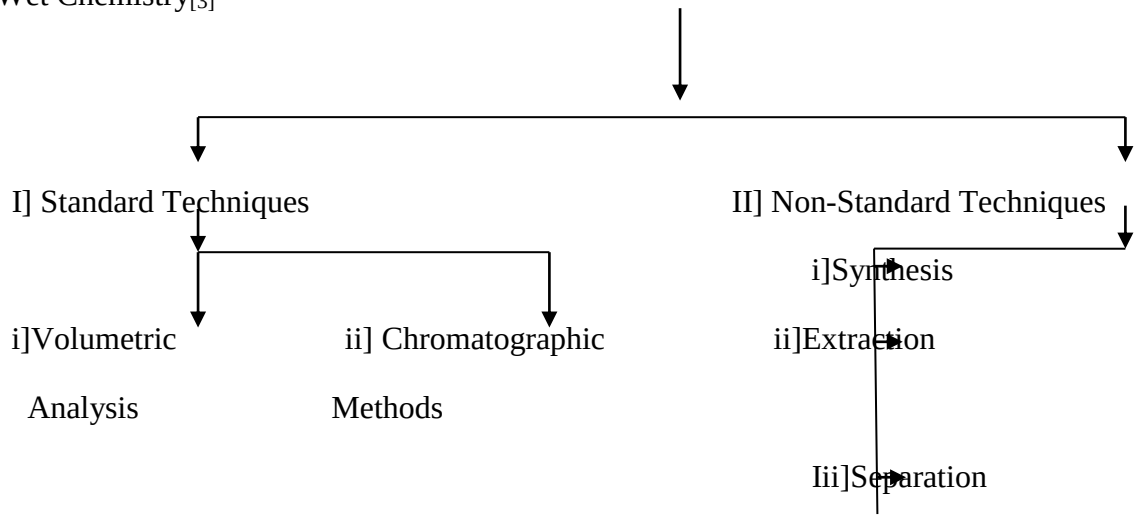
Example-

1] Cleaning of bathroom- In this Tetra-clean white phenyl concentrate is measured 25 ml and diluted with 1000 ml (1 Liter) water(H_2O).

2] Acid wash carried out for cleaning tiles- In this 28ml HCl is diluted with 1000 ml (1Liter) water(H_2O).

The classification of wet chemistry is as follows-

Wet Chemistry[3]



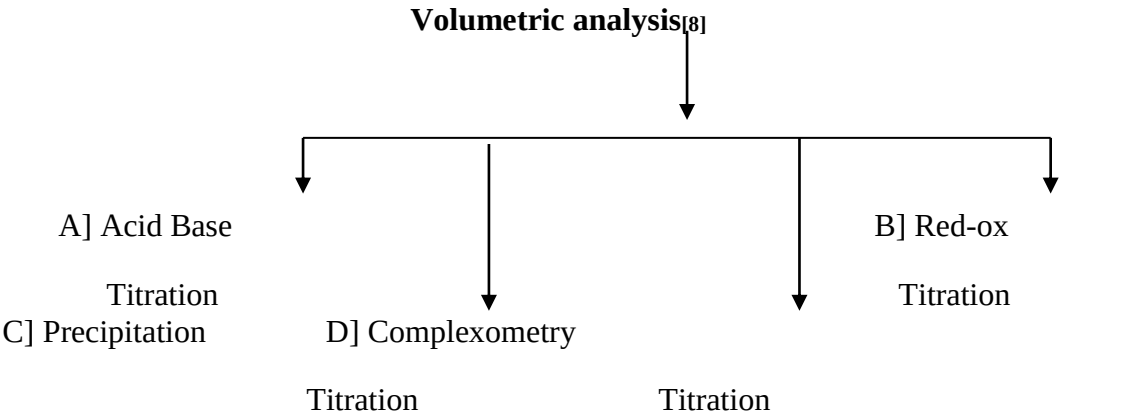
I] Standard Techniques-

i] Volumetric analysis -

The word volumetric means 'volume'. The volumetric analysis determines how much volume is consumed during analysis process. Volumetric analysis mainly includes titration by sing indicators like phenolphthalein, methyl orange, etc. which results color during titration which is a sign that reaction has ended or this is the end point of titration.

Sometimes there is no need to add the indicator as some reagents acts as a self indicator. Example- KMnO4 (Potassium permanganate) acts as a self indicator.

The volumetric analysis is as follows-



A] Acid Base Titration[4,8]

The process of adding titrant to titrate is called titration. Titrant means 'a substance (reagent) of known molarity (Concentration)' Titrate means 'to verify the quantity of given constituent by adding titrant to another constituent of known concentration until the concentration reaches neutralization.

Acids have pH less than 7 and base have pH more than 7.Acid is a chemical that that donate proton and accept the electron.Base is a chemical that accept proton and donate electron.Example of acid- HCl(Hydrochloric acid)Example of base- NaOH(Sodium Hydroxide)Classification of Acid base titration is given below[7]

1)Strong acid & Strong base - HCl & NaOH

2)Strong acid & Weak base – HCl & NH3

3)Weak acid & Strong base – CH3COOH & NaOH

4)Weak acid & Weak base – CH3COOH & NH3

One experiment which is commonly carried out at schools which involves acid base titration-

15 ml 0.1N HCl taken in conical flask followed by phenolphthalein indicator (5 to 8 drops) which is titrated with 0.1N NaOH (present in burette) where pink color appears it is considered to be the end point of the reaction. This is simple titration.

There is also an experiment called 'back titration' or 'reverse titration' in which HCl is present in burette and in conical flask NaOH is present. When Phenolphthalein indicator is added to flask the pink color appears at initial before titration. When titration is carried out using burette (HCl) solution the pink color disappears. The point at which the pink color disappears is considered as an end point of the reaction.

B]Red-ox Titration [9]

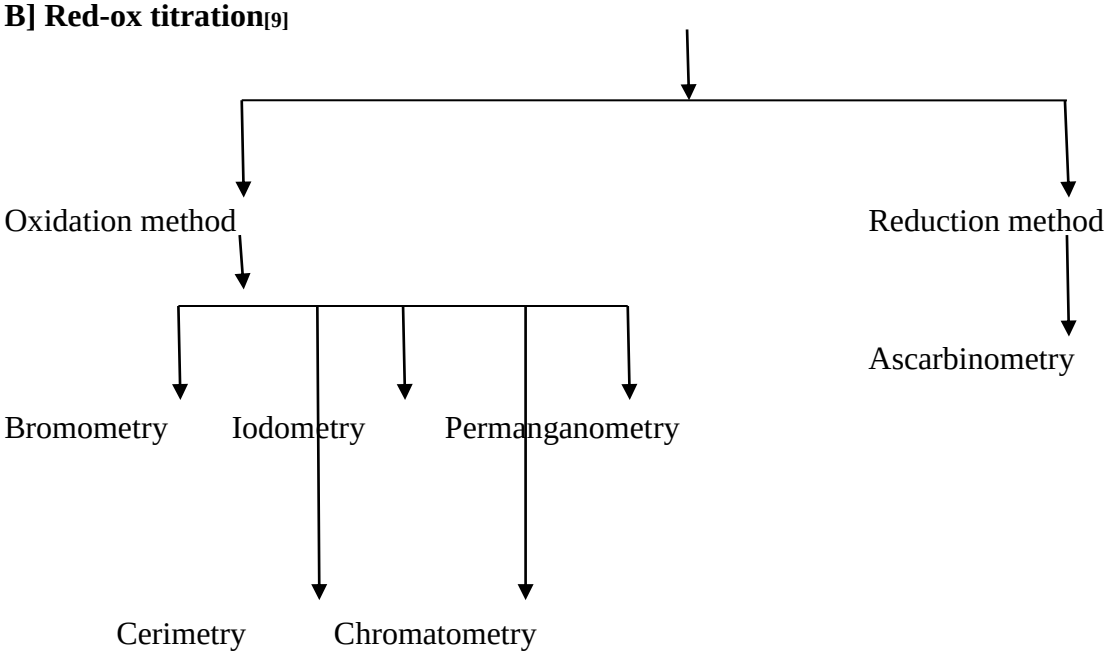
The word 'Red-ox' can be meant as 'Red' which means 'reduction' and 'Ox' which means 'oxidation'. The word reduction means addition of hydrogen or removal of oxygen in a reaction.The word Oxidation means addition of oxygen or removal of hydrogen in a reaction. [8]

As mentioned earlier about indicators, in the red-ox type of titration there are some reagents which acts as a self indicator while in some reagents there is need to add the indicator.

The self indicator example are – $KMnO_4$ while external indicators (which are added to the reagents while titration) are 'starch indicator', 'methylene blue', etc .

Classification of red-x titration is as follows-

B] Red-ox titration[9]



Explanation of classification –

As shown above this is the classification of 'Red-ox' type of titration which is sub category of volumetric analysis. The 'Red-ox' titration is classified into two main classes viz-

a)Oxidation method

b)Reduction method

The oxidation method is sub classified into five classes viz-

i. Bromometry

ii. Iodometry

iii. Permanganometry

iv. Cerimetry

v. Chromatometry

The reduction method is sub classified into one class viz-

i. Ascarbinometry

Some reagents used for titrations like-

For Bromometry potassium bromated is used .

For Permanganometry potassium permanganate is used .

For Ascarbinometry ascorbic acid is used .

Example of red-ox titration[8,9]

$3H_2C_2O_4 + 2KMnO_4 \xrightarrow{} 2H_2O + 6CO_2 + 2MnO2 + 2KOH$

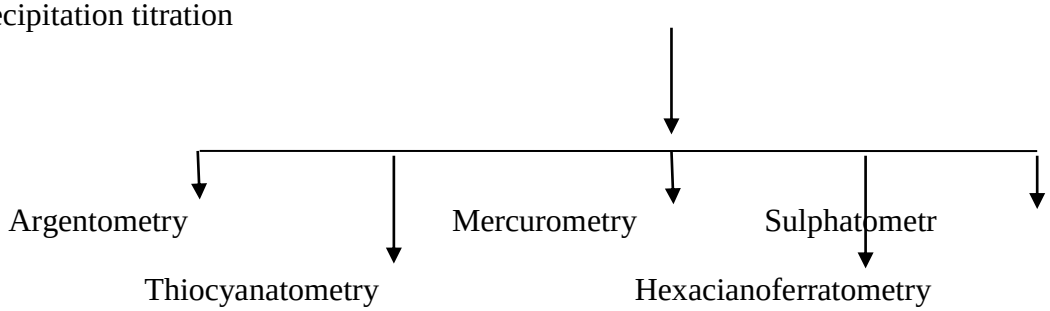
(Oxalic acid) (Potassium Permanganate) (Water) (Carbon dioxide) (Manganese (IV) Oxide) (Potassium Hydroxide)

C]Precipitation Titration[10]

In this type of titration precipitation agent like (CaCO3) are used as a titrant.

Classification of precipitation titration-

Precipitation titration



As like other titrations in this method, during titration at the end point there is formation of precipitation .The most commonly used indicator used in this type of titration is potassium chromate.

The commonly experiment which is carried out at the school level about precipitation titration is AgNO3 when reacts with Cl^- ions after the precipitation / completion of reaction there is formation of AgCl and NO_3-

D] Complexometry Titration [6]

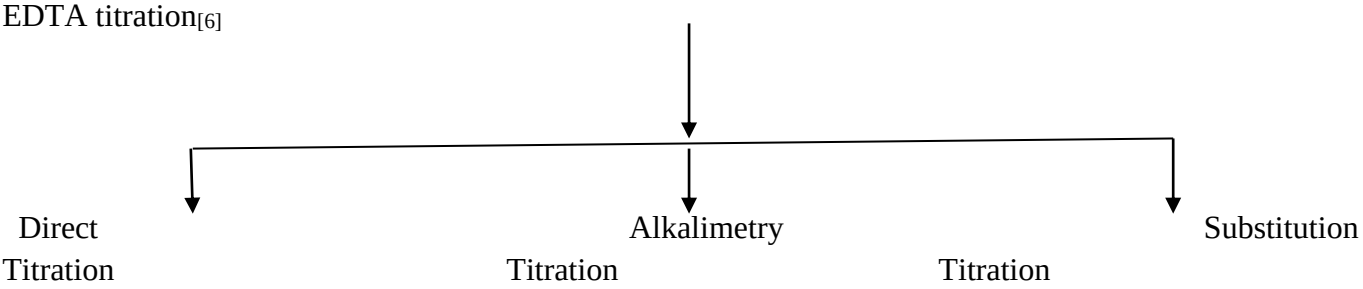
Complexometry titration also called as 'Chelatometry' is a type of volumetric analysis.

In this type of titration the colored complex are formed which is used to determine as an end point of titration.

The another name for Complexometry titration is EDTA; Ethylene Diamine Tetra acetate.

Classification of EDTA Titration-

EDTA titration[6]



There are few indicators used in the Complexometry titration viz8

Erichrome black T indicator

Xylenol indicator

Hydroxynapthol Indicator

EDTA titration follows the fact that the metallic ions present in the chemicals forms the complex with tetrahedral ligand for the stability of complex.

Example-

$Cu^{2+} + 4(NH3) \longrightarrow Cu(NH3)_4$

ii] Chromatographic methods[11]

Greek words chroma (meaning "color") and graphein (meaning "to write") are the sources of the word "chromatography." This method involves separating the mixture on a stationary phase, which can be either liquid or solid. A pure solvent, like water, or any other gas, is allowed to pass slowly over the phase that is stationary.
The process of chromatography involves combining the analyte with a fluid or gas mobile stage and passing it via a phase that is stationary. Typically, there are two phases: one lipophilic and the other hydrophilic. With these two phases, the analyte's constituent parts interact in various ways. Particles invest more or fewer seconds engaging with the stationary phase, based on their polarity. This leads to each of the parts found in to be segregated.

Chromatography can be classified as follows-

Chromatography

↓

Seperation / Identification Phases

A] Thin Layer Chromatography(TLC)

B] Paper Chromatography

C] Column Chromatography

1) Stationary Phase[6,11]

A] Thin Layer Chromatography (TLC)-

The technique of Thin Layer Chromatography(TLC) is employed to separate combinations that are not volatile. The experiment is performed on a piece of glass, plastic, or aluminum foil that has been lightly coated in an adsorbent compound which typically consist of silica gel, cellulose, or oxides of aluminum are the materials being employed for the stationary phase.

Thin-layer chromatography (TLC), like other techniques for chromatography, is based on the splitting principle. The relative affinities of the chemicals for the two phases determine the separation. Across the outer layer of the stationary phase, the compounds in the phase of mobility travel. The compounds that have a stronger affinity for the stationary phase migrate slower than the other compounds, with the

movement happening in this way. As a result, the mixture has been separated. The mixture's constituent parts emerge as spots at their appropriate levels on the plates once the separation procedure is complete.

The Whitman Filter paper can also be used for this type of chromatography.

Example of TLC –

Garlic extract TLC was performed .

In this process, test solution used – Garlic extract

Stationary phase- Silica Gel G

Mobile phase- Methanol & water (1:1)

B] Paper Chromatography[2]

Compounds are able to be sorted via paper chromatography based to their shifting solubility in both the stationary and mobile phases (solvent).

Paper chromatography is an approach of chromatography in which sheets of paper or bands act as the adsorbent, or stationary phase, by which the liquid is allowed to move through.

The paper chromatography is based on the principle of partition methodology. Paper chromatography consist of two phases viz, the stationary and the mobile phase.

Within paper chromatography, H_2O acts as stationary phase whereas solvent rather than H_2Oacts mobile phase.

Example of Paper chromatography-

In this process, Standard sample used was D-Glucose, and test sample to other side.

Stationary phase – Silica Gel G

Mobile phase- Butanol, Acetic acid, Water (4:1:5)

C] Column chromatography[12]

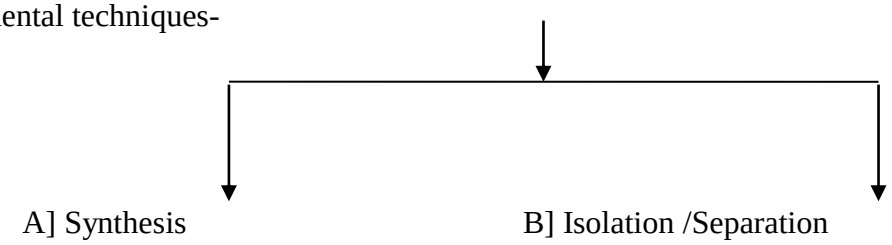
The word column means long tube. Separation of material / compounds which are processed in glass long tube filled with stationary material i.e. Stationary Phase is called column chromatography. The sample mixture is filled into the column & is sealed with the stationary phase and permitted to move through the glass column. This causes to segregate the components as per the affinity towards lipophilicity & hydrophiliocity phases present within glass column.

II] Non- Standard Techniques

The non-standard techniques are those which do not involve the use of modern methods instruments. The non-instrumental techniques requires only glassware apparatus.

The non-instrumental techniques involves –

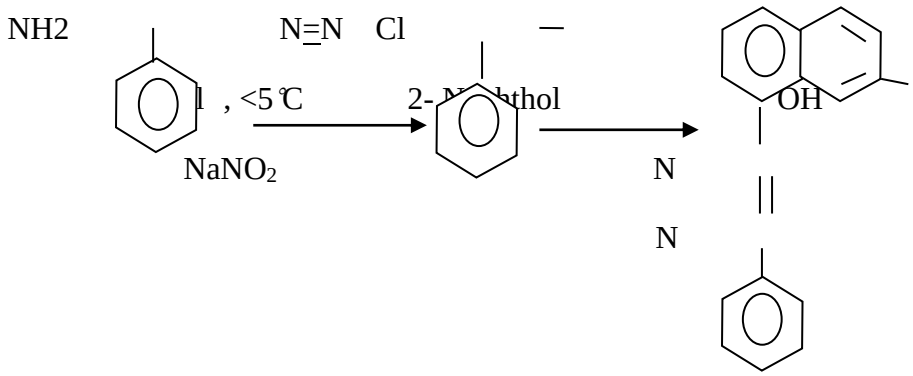
Non-instrumental techniques-



A] Synthesis[13]

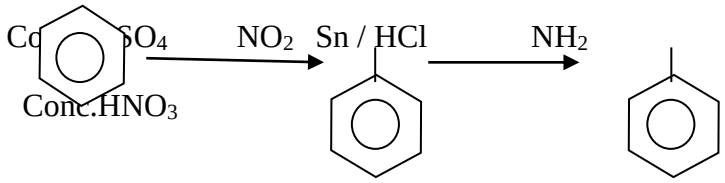
Many of the reactions in wet chemistry follow the solid- liquid, liquid- liquid synthesis. Synthesis i.e. formulating of new compounds or substance is mostly carried out using non- instrumental techniques . The non-instrumental techniques consist of glassware apparatus like RBF, beaker, china dish, etc. The synthesis can be carried out using solid cruds drug , as well as liquid (Wet) chemicals.

The best example of synthesis of Azo dye is given below – [13]



$C_6H_5NH_2$ (Aniline) when reacts with HCl & $NaNO_2$ where, temperature is maintained at 5˚C, it initiates the diazotization reaction which gives intermediate. This intermediate further reacted with 2-Naphthol it initiates coupling reaction which gives and end product called Azo Dye . The Azo Dye is red or cherry red in color which is widely used in food industry, leather industry, pharmaceutical industry. This Azo dye synthesis is made using Solid-Liquid interactions/ chemicals.

Another example of synthesis using wet chemicals (Liquid-liquid) includes – [14]



In a RBF, Benzene is taken as per required quantity mentioned in handbook. When C_6H_6 reacts with Conc.H_2SO_4 and Conc. HNO_3 it gives an liquid intermediate called Nitrobenzene . Further when nitrobenzene undergoes clemmenson condensation (containing Sn/ HCl) and 6(H) molecules, it gives end product called Aniline .

B] Isolation / Separation

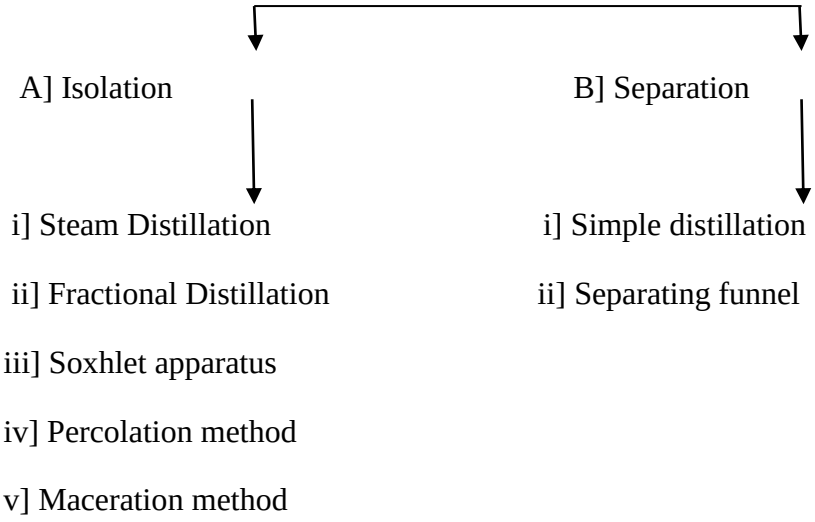
Moving towards isolation and separation, as in these method or processes both terms called isolation and separation are different accordingly-

Isolation-

Also called as extraction in which the liquid is used as reagent and heat or pressure is applied so as to get the constituents present in the crude drug which can present in the form of wood, leaf, flower, etc.

Separation-

Also called as parting in which the active constitutions or the liquids (Miscible or immiscible) can be separated with the help of distillation assembly or eparating funnel based on their densities.



A] Isolation Method[15]

The isolation method/ technique is used mostly for the isolating purpose . The isolation method carries out the extract of active constituents from the crude drugs. The equipments used for this type of method are steam distillation, Fractional distillation, Soxhlet apparatus, Percolation method and Maceration method.

Example of such method is discussed below –

Isolation of curcumin from Haldi or Haridra –[15]

25gm of powdered dried turmeric rhizome is taken in a large beaker and mixed with 1000ml of distilled n-hexane using a magnetic stirring rod. The resultant suspension is stirred for 3 days, then filtered and placed in a "thimble" of Soxhlet apparatus.310ml of methanol is heated in a flask, and its vapours are condensed in a condenser. The condensed extractor drips into the thimble containing powdered turmeric rhizome, and extracts it. When the liquid level in chamber rises to the top of siphon tube, the liquid contents flood into the flask. This process is continuously carried out till a drop of solvent from the siphon tube does not leave residue when evaporated. After 3 days, the solvent is evaporated and dissolved in 100ml of toluene and then the following solution obtained is poured into a separating funnel, added with 100ml 0.2M NaOH, and shaken for a few minutes. The aqueous phase is collected and

acidified to pH 3 using 0.2M HCl. The brown extract turns yellow as it undergoes clarification. The filtrate is extracted with diethyl ether (3×100 ml). In the final extraction, the ether turns pale yellow colored indicating the end of extraction. The combined ethereal phases are washed with 30ml water and dried over $MgSO_4$. Ether is removed completely under vacuum to leave a yellow colored solid crude curcuminoid, which is purified by thin layer chromatography.

B] Separation Method[15]

In this separation method the liquid (Wet) compounds can be separated easily. The miscible and immiscible both liquids can be separated using this method. For separation of miscible liquids the simple distillation assembly can be used. Whereas for the separation of immiscible liquids separating funnel can be used.

Take an example of separating miscible mixture –

Acetone and water which are to be separated . Thus the boiling point of acetone is 56°C where as the boiling point of water is 100°C. This miscible mixture is then poured or added to RBF and the RBF Assembly is setuped by joining the condenser, inlet-outlet pipes, receiver, etc. Now apply the heat to the bottom of the RBF . Note that temperature do not rise above 60°C. This will lead to boil the acetone and the gaseous fumes will rise up and pass through cooled condenser and will get convert from gaseous state to liquid state. This is how miscible liquids can be separated. Also the other distillation assembly can be used like Steam distillation, fractional distillation but the result are not obtained at that height.

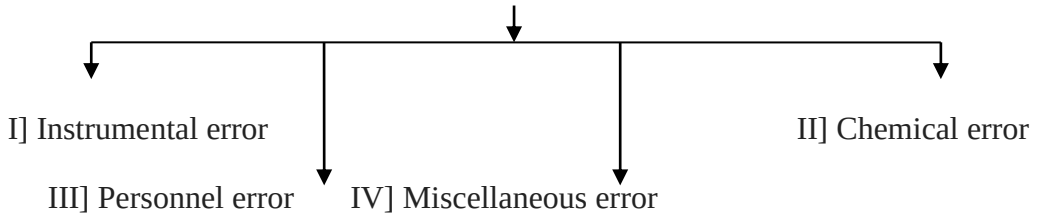
Take an example of separating immiscible liquids -

Hexane and water are to be separated . This immiscible liquids can be separated by using separating funnel. The mixture of hexane and water are added to the separating funnel. Allow to stand the separating funnel on tripod stand for at least 10 minutes. Then slowly move or rotate the knob of separating funnel and collect the liquid of higher density in one beaker . Then shut the knob. Take another beaker and into it , rotate the knob and into the another beaker collect the lower density fluid .In this way the immiscible liquids can be separated.

Another example which can be included into immiscible type of separation is Partition coefficient where the partition coefficient test is performed using Hexane and Water or Octane and Water.

Errors encountered in wet chemistry[6]

Errors



I] Instrumental error-

The instrumental error consist of the error which arise in the glassware and modern techniques instruments . The error which arise in glassware can be as follows-

Breakage of glass apparatus, unwashed or unsterilized glassware, modern apparatus use for extraction can undergo maintenance, etc . Also the improper glassware used for the experiment , improper followed experiment techniques during experimental work can lead to error.

II] Chemical error-

The Chemical error consist of the error which arise in the chemicals and into their concentrations. . The error which arise in chemicals can be as follows- The concentration of any chemical viz HCl , if found wrong or incorrect during experiment it can lead to great error. Also the normality or molarity conversions calculation if went wrong it can lead to error. The concentrations of reagent if went wrong can lead to error. Also if the industry preparation of chemicals is of less concentration, the wanted result will not be gained.

III] Personnel error-

Personnel error can occur when handling of chemicals , apparatus , instruments in the improper way. This can lead to change in the net result which aren't obtained and can cause error . The personnel error involves hygiene, improper apparatus handling, improper sterilization, improper handling of chemicals, improper chemical usage,etc.

IV] Miscellaneous error-

Miscellaneous error involves the environmental factors, following wrong SOP, un-following given commands in experimental rooms, un-following standard procedure in experiment, keeping chemical bottles at unsafe places, etc. Keeping chemicals at the unsafe place near burner or in the contact of direct sun, withdrawal of chemical from bottles using unsterilized glassware, using tap water or undistilled water, etc can also lead to error.

Glassware used in wet chemistry-[15,16]

The glassware used in chemistry, Wet chemistry practical, experimental work are not a normal glass used. These glass are of special type. At most care is taken on & after experimental work. These glasses should be of special type only. Few glasses are liable to use for experimental work, while some are not, while some are used for keeping chemical liquids, while some only for powder substances and not for liquids. The glasses or glassware used for experimental work are of four types viz,

Sr.No	Type	Category	Uses-
1	Type I	Highly resistant borosilicate glass	Used for all liquids and powders of any pH
2	Type II	Water attack test	For buffer aqueous solution with pH below 7 & for dry powders
3	Type III	Sodalime glass	For dry powders and not for liquids
4	Type IV	General purpose sodalime glass	Not used for any chemical liquids

Conclusion-

Wet Chemistry is the one subject that involves Pharmaceutical Analysis, Pharmaceutical Chemistry, Organic Chemistry, Inorganic chemistry . The techniques used in wet chemistry are beneficial in the field of titration, synthesis, assay, identification , separation, extraction of new drug/ compound, etc. Wet chemistry not only applies the fundamentals of Chemistry but also allows the experimental fellows to carry out the wide and wast ways in the organic, inorganic, Analysis field.

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