Quantitative and Qualitative Analysis

Quantitative analysis is an analysis method used to determine the number of elements or molecules produced during chemical reaction. Organic compounds are comprised of carbon, hydrogen, oxygen, nitrogen, phosphorous, sulphur and halogens.
While the Qualitative analysis is a method to analyze the species present in a compound and is more focused on finding the elements ~~present a~~ and ions present in the given compound.

How do you analyze organic Compound?

The first one is the use of chemical tests to identify the functional groups. The second aspect is the use of high resolution mass spectrometry to identify the mass and bonding positions. The third one is the use of IR spectroscopy to confirm the functional groups and other bonding modes through "fingerprinting".

(A) Preliminary Identification Tests

(1) Colour and physical State: - These have important role to play in preliminary identification of organic compounds. For examples, Naphthol are usually pink in colour and ~~aos~~ most of them are solid in nature; Nitro compounds (eg. Nitrobenzene) are yellow in colour. Some colourless liquid organic compounds includes alcohols, aldehydes, ketones, simple esters, lower ethers and aromatic hydrocarbons. The colour and physical state of some organic compounds may change on long storage due to climatic and environmental factors.

(2) Odour :- The odour of organic compounds also help in their identification. For example, Amines possess fish-like smell, Nitrobenzenes possess almond-like smell, acetamides, and acetonitriles possess rat-like smell, etc. The odour compounds may change with time due to storage and environmental factors.

(3) Solubility: Solubility of organic compounds also helps in identifying if the compound is acid, or basic or neutral. Some of the solutions used to check the solubility are water (H_2O), dilute Hydrochloric acid (HCl), dilute Sodium Hydroxide ($NaOH$) and dilute Sodium Carbonate (Na_2CO_3). Water is used to check the Solubility by pH analysis of the dissolved compounds in order to determine the pH scale of the solution, whether it is acidity (1 - 6.9), basicity (7.1 - 14) or neutral (7.0). Acidic Compounds are insoluble in HCl, Soluble in $NaOH$ and produces effervescence with Na_2CO_3 while basic compounds are soluble in HCl, insoluble in $NaOH$ and do not produce effervescence with Na_2CO_3. Neutral Compounds are either soluble or insoluble in HCl and $NaOH$.

(B) Extraction

This is a method of separation that utilizes the differences in solubility of materials present. Extraction process depends on chemical and physical characteristics of the targeted compound, availability of the plant sample, eco-friendly approach and yield of the extract. There are different extraction methods which include :...

(1) Reflux Extraction: Used for extraction of volatile components but not for thermolabile natural products and it is ~~more~~ efficient ~~than~~ and requires less time and solvent.

(2) Soxhlet Extraction: It is an automatic and continuous extraction method which utilizes the principle of reflux and siphoning to continuously extract sample with fresh solvent. It is an efficient method of extraction and requires a lot of solvents.

(3) Pressure Liquid Extraction: It uses high pressure to keep solvent liquid above boiling point for extraction of lipid solutes - which has low solubility and diffusion. It has efficient solvent and less time utilization.

4) Pulse Electric Field Extraction:– It destroys membrane structures to encrease mass transfer using non-thermal, pulse field strength and specific energy for extraction.

5) Supercritical Fluid Extraction:– It is used to extract the thermolabile components using supercritical fluid (eg S-carbondioxide) with good solubility and diffusivity to liquid and gas respectively.

6) Simple Distillation Method:– This is used for separation of liquids (miscible) based on the differences in their boiling points whereby the substance with lower boiling point evolves as the boiling temperature is reached before the substance with higher boiling point.

(C) Chromatography

This is a biophysical technique which enable the separation, identification and purification of compounds present in mixture of a qualitative and quantitative analyses. The compound to be seperated are distributed into two phases; the stationary and mobiles phases.

The two types of chromatography are – (a) The Column Chromatography which is used in High performance Liquid Chromatography (HPLC), Gas Chromatography (GC), etc

(b) Planar Chromatography which is used in paper chromatography, Tin-Layer Chromatography, etc.

The Functional Group Tests & Analysis

(1) Test for Alcohols

Alcohols are organic compound containing hydroxyl (-OH) functional group. Organic alcoholic compounds containing one -OH group are termed as monohydric, dihydric for two -OH groups and trihydric for three -OH groups. They are further classified as primary (CH_3CH_2OH), Secondary ($CH_3CHOHCH_3$) and tertiary ($CH_3COHCCH_3CH_3$) alcohols based on the respective number of the R-groups attached to the carbon atom bearing the hydroxyl group.

Example

Iodo Form Test

Materials required:- Organic Compound, 1% iodine solution, dilute NaOH, test tube, droppers and water bath (use beaker of water placed on a tripod stand under Bunsen burner flame as an alternative).

Procedure:

(1) Take small quantity of organic compound into a clean test tube.
(2) Add few drops of 1% iodine solution using a dropper.
(3) Add dil. NaOH dropwise until the brown colour of iodine disappears
(4) Warm the content mixture gently in a water bath.

Result

A yellow precipitate of iodoform is formed.

(a) $CH_3CH(OH)CH_3 + I_2 + NaOH \longrightarrow CH_3(CO)CH_3 + NaI + 2H_2O$

(b) $CH_3(OH)CH_3 + 3I_3 + 4NaOH \longrightarrow \underset{\text{Iodoform}}{CHI_3} + CH_3COONa + 3NaI + 3H_2O$

(2) Test for Phenols

These are compounds containing one or more hydroxyl groups attached to aromatic ring. Examples are Cresol, Catechol, etc. They are usually white crystalline solid.

Examples

(a) Litmus Test

Materials required: Organic Compound, moist blue litmus paper, Spatula and watch glass.

Procedure:

Take a small quantity of organic compound and place on a moist blue litmus paper placed on a watch glass.

Result:

Phenol is a weak acid and it turns the colour of the moist blue litmus paper red.

(b) Ferric Chloride Test

Materials required: Organic compound, ferric chloride (neutral) solution, spatula, test tube and droppers.

Procedure

(a) Take small quantity of neutral solution of ferric chloride onto a clean test tube.

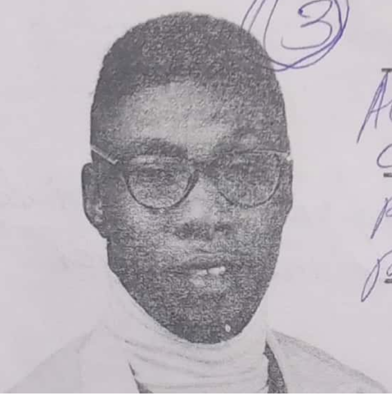
(b) Add small quantity of organic compound onto the solution.

Result:

- Formation of a violet colour of a complex.

$$C_6H_5OH + FeCl_3 \longrightarrow Fe[(OC_6H_5)_6]^{3-} + 3HCl\ 3H^+$$

(3) Test For Aldehydes



Aldehydes are organic compounds in which the carbonyl carbon is attached to hydrogen and R-group (R = alkyl or aryl group). Aldehydes are presence in almond, cinnamon, etc.

Example

(a) 2, 4 - Dinitrophenyl hydrazine test

Materials required:- Organic Compound, rectified spirit, 2, 4-dinitrophenyl hydrazine solution, test tube and droppers.

Procedure:

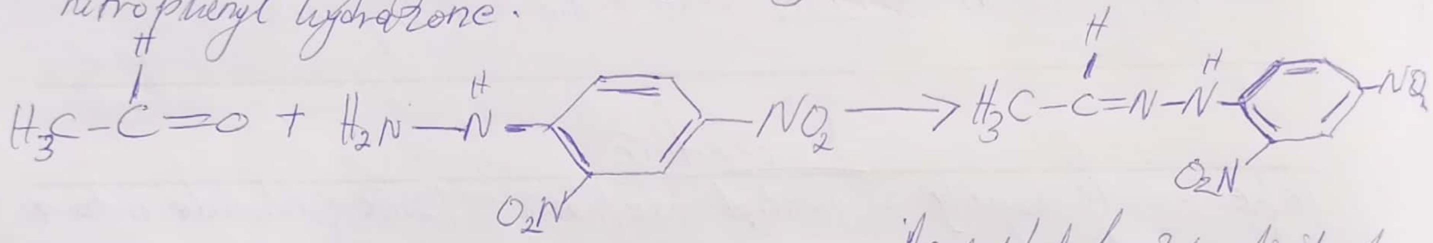
(a) Take a small quantity of organic compound into a clean test tube.

(b) Add few drops of rectified spirit using a dropper and shake well.

(c) Add few drops of 2, 4-dinitrophenyl hydrazine solution and shake well.

Result

- Formation of a yellow or ~~org~~ orange precipitate of 2, 4-dinitrophenyl hydrazone.



Acetaldehyde-2,4-dinitrophenyl hydrazone

(4) Test for ketones

Ketones are organic compounds containing carbonyl carbon that is attached to R-groups, that is, R and R' groups. The R-groups can be alkyl or aryl groups. The presence of ketones is observed on the flavours of berries, mushrooms, etc.

Example

Sodium Nitroprusside Test

Materials required: Organic compound, sodium nitroprusside crystal, dilute NaOH, distilled water, test tube and droppers and spata.

Procedure

(a) Take a small quantity of the crystals of sodium nitroprusside into a clean test tube using a spatula.

(b) Add small amount of distilled water and shake well untill the crystal is dissolved.

(c) Add small quantity of organic compound using a dropper.

(d) Add few drops dillute NaOH using another dropper and shake.

Result

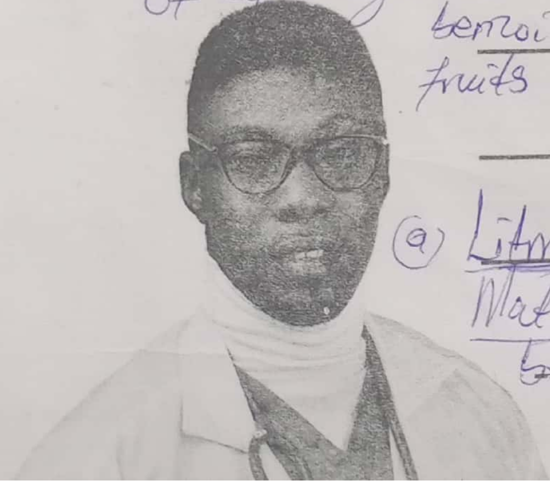
- Formation of red coloured complex.

(a) $\underset{\text{Ketone}}{CH_3COCH_3} + OH^- \longrightarrow \underset{\text{ketone anion}}{CH_2COCH_2^-} + H_2O$

(b) $\underset{\text{(Nitroprusside ion)}}{[Fe(CN)_3NO]^{2-}} + CH_2COCH_2^- \longrightarrow \underset{\text{(red coloured complex)}}{[Fe(CN)_3NO \cdot CH_2COCH_2]^{2-}}$

(5) Test for Carboxylic Acids

They are organic compounds containing carboxyl functional group (–COOH). Aliphatic and aromatic carboxylic acids are two types of carboxylic acid. Examples are acetic acid, formic acid and benzoic acid, etc. Carboxylic acids are found in fruits such as orange, apple, lemon, etc.



Examples

(a) Litmus Test

Materials required: Organic compound, moist blue litmus paper, dropper and watch glass.

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Procedure:

- Take few drops of organic compound using a dropper onto a moist blue litmus paper placed on a watch glass.

Result

- The blue litmus paper turns red, showing it is acidic in nature.

(b) Sodium Bicarbonate Test

Materials required: Organic compound, sodium bicarbonate, spatula, test tube and dropper.

Procedure

(a) Take small quantity of organic compound into a clean test tube using a dropper.
(b) Add small quantity of sodium bicarbonate to the compound using a spatula.

Result

- Brisk effervescence of Carbon(IV) oxide gas is produced.

$$RCOOH + NaHCO_3 \longrightarrow RCOONa + H_2O$$

~~(b) Test~~

Qualitative Analysis

In the course of experiment, chemical solutions of compounds are calculated and prepared to the concentrations required before they are used for tests, reactions and analyses.

(1) Preparation method of solution in distilled water using solid chemical compound.

Procedure is as follows:

(1) Calculate the amount of solute required in 1 liter of the solvent (distilled water or deionized) at the required molarity.

(2) Weigh out with watch glass using a weighing balance.

(3) Transfer to a calibrated beaker containing half (0.5L) volume of the solvent.

(4) Wash of the remaining particles of the solute attached to the watch glass with few volume of the solvent ~~of~~ ~~[illegible]~~ is

(5) Stir with a stirrer (glass rod).

(6) Apply small heat to assist fast dissolution in case of delay dissolution.

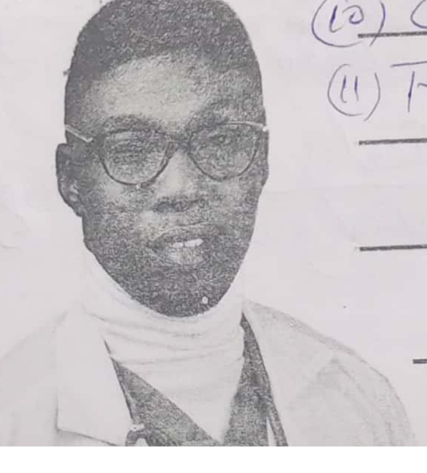
(7) Transfer the content of the beaker to a volumetric flask or a measuring cylinder using glass funnel.

(8) Rinse the beaker, glass rod and glass funnel into the flask with few amount of the solvent.

(9) Make up the solution in the flask with the solvent up to 1 Liter mark (at the meniscus).

(10) Cork flask with a stopper and mix thoroughly.

(11) Transfer to the labelled reagent bottle.



CONGRATULATIONS

(2) Determination of Concentration by Percentage of Substances

This can be expressed as percentage Weight/Volume (% W/V) or percentage volume/Volume (% V/V) or percentage weight/weight (% w/w).

(a) For Solid Solute:

Percentage weight per volume (% w/v) is used and expressed as the mass of solute (solid) in grams dissolved in 100ml of solution.

$$\% w/v = \frac{\text{Mass of solute (g)}}{\text{Volume of Solution (mL)}} \times 100\%$$

(b) For Liquid Solute:

percentage volume per volume (% v/v) is used and expressed as the volume of solute (Liquid) in milliliter per 100 mL of solution.

$$\% v/v = \frac{\text{Volume of Solute}}{\text{Volume of the solution}} \times 100\%$$

Note that for Weight Percentage (% W/W):

this is the mass of solute (in grams) and in (100g) of solution. The solution can be prepared independent of the temperature considerations, usually in a Commercial preparation.

$$\% W/W = \frac{\text{Mass of Solute}}{\text{Mass of Solution}} \times 100\%$$

(3) Determination of Percentage Yield

In the course of an experiment, the yield or product in percentage is calculated using the practical yield against the theoretical yield. It is assumed that the materials used in the practical react in such a way that the quantity expected as product of the reaction may decrease from the stoichiometric quantity evaluated.

$$\% \text{ yield} = \frac{\text{Practical (g)}}{\text{theoretical yield (g)}} \times 100\%$$

$C = \frac{n}{V}$

$\therefore n = CV$

(4) Preparation of Solution by dillution

In dillution, more solvent (dyluent) is added to a given solution. Solutions can be prepared by dillution. A solution of higher concentration is dilluted to produce solutions with lower concentrations. A stock solution is a concentrated solution that is dilluted to a lower concentration, also called working solution.

The dilution does not alter the number of moles of solutes present as they are the same before and after the dilution but alters only the final volume.

Concentration of Solution (C) in mol/L

$$= \frac{\text{number of moles of the solute } (n)}{\text{Volume of the solution } (V) \text{ in Liter}}$$

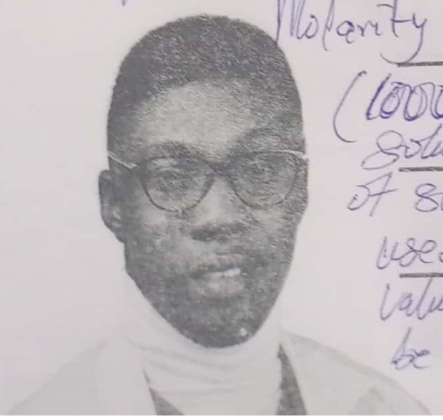
$$\therefore n = CV$$

Therefore, for the two dillutions: initial dilution (1) which is the concentrated solution and final dilution (2) which is the diluted solution:

$$C_1V_1 = C_2V_2$$

$$V_1 = \frac{C_2V_2}{C_1}$$

(5) Preparation of a Molar Solution



Molarity is the number of moles of solute per liter (1000 mL) of solution. In preparation of saturated solution of a liquid solute, the number of volume of solvent (usually distilled or deionized water) to be used in such preparation is calculated. The known values required are the molarity of the substance to be prepared (M), specific gravity or density, percentage ~~yield~~ assay, and molecular mass. The figures are usually written on the body of the container of the commercial chemical product.

For example, you should know that an average concentrated Hydrochloric acid (HCl) has a molecular mass of 36.5 g/mol, specific gravity of 1.18, and percentage assay of 36%.

Mathematically,

$$\text{Volume in } 1L \text{ for } 1M = \frac{\text{Concentration (M)} \times \text{molecular mass (g/mol)}}{\text{percentage assay} \times \text{specific gravity}} \times 100\%$$

Note that most liquid concentrated chemical solutions have their molecular mass, specific gravity and percentage assay data written on the bottle or container labels