UNIT 2A3 - PAPER 2

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UNIT 2A3 - PAPER 2

BASIC PRINCIPLE OF THERMAL ANALYSIS AND APPLICATION

1. TGA –Thermogravimetric Analysis
2. DTA – Differential Thermal Analysis
3. DSC – Differential Scanning Calorimetry

UNIT - 2A | PAPER - 2

ANALYTICAL CHEMISTRY AND STATISTICAL TREATMENT OF ANALYTICAL DATA

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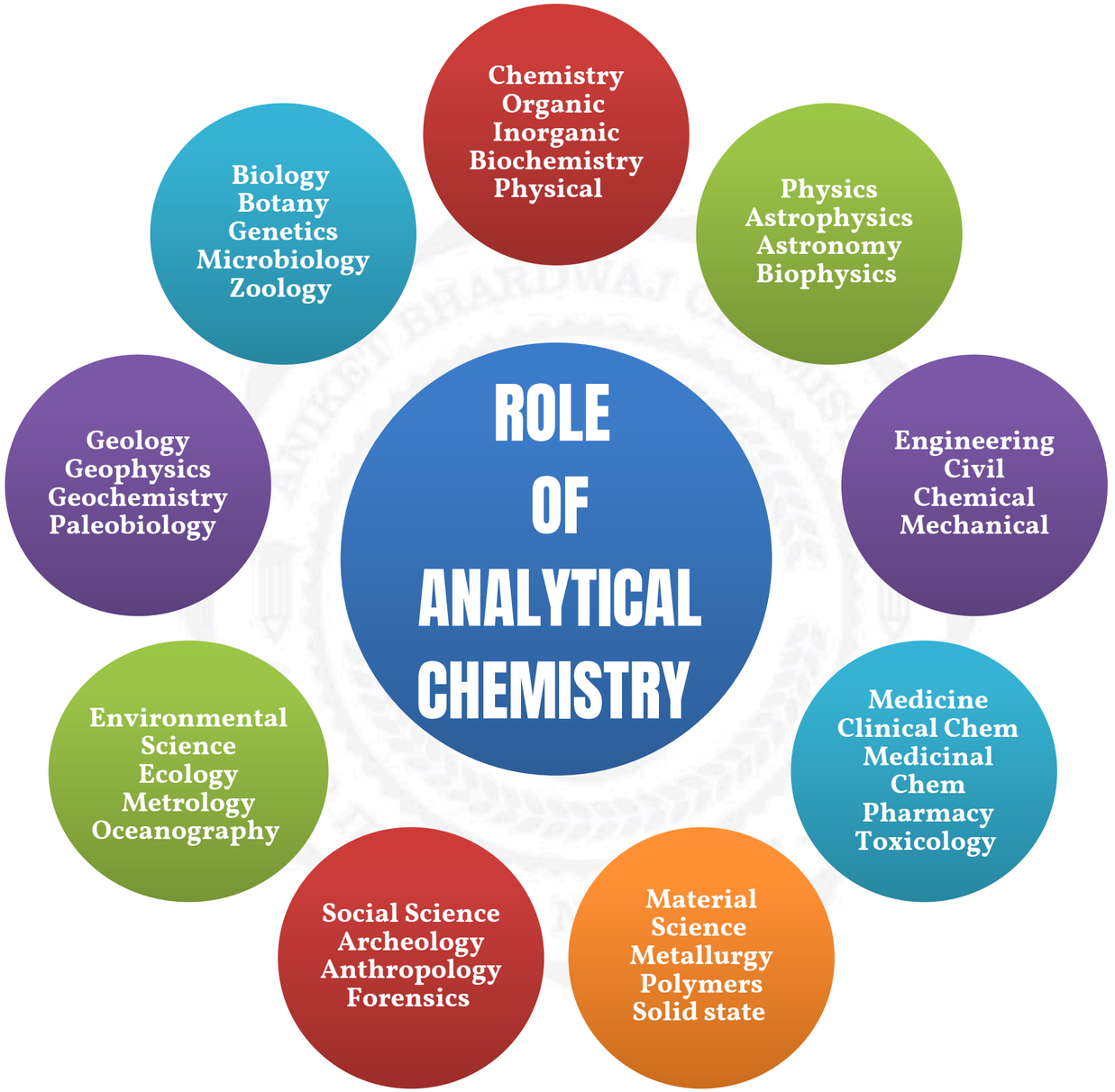


THE LANGUAGE OF ANALYTICAL CHEMISTRY

1. **Analysis:** A process that provides chemical or physical information about the constituents in the sample or the sample itself.
2. **Analyte's:** The constituents of interest in a sample.
3. **Matrix:** All other constituents in a sample except for the analyte's.
4. **Determination:** An analysis of a sample to find the identity, concentration, or properties of the analyte.
5. **Measurement:** An experimental determination of an analyte's chemical or physical properties.

THE LANGUAGE OF ANALYTICAL CHEMISTRY

6. **Accuracy:** The agreement between measured value and True value

7. **Precision:** The Agreement between number of Observations

8. **Selectivity:** It is the degree to which the method is free from interferences' present in matrix.

9. **Sensitivity :** Ability of a method to discriminate between two small concentration differences in the analyte

10. **Selectivity:** It is defined as degree to which the method is free from interferences from other components present in the matrix.

THE LANGUAGE OF ANALYTICAL CHEMISTRY

I1 **Dynamic range:** It is the concentration range from limit of Quantification (LOQ) to Limit of Linearity (LOL).

I2 **Limit of Linearity (LOL) :**
It is defined as maximum concentration range up to which instrument is produces linear response.

I3 **Limit of detection(LOD):**
Minimum amount of concentration of a component that can be detected with a given degree of confidence.

I4 **Limit of Quantification (LOQ):**
Minimum amount of concentration of a component that can be estimated with a given degree of confidence is termed as LOQ.

THE LANGUAGE OF ANALYTICAL CHEMISTRY

15 **Technique:** A chemical or physical principle that can be used to analyze a sample.

16 **Method:** A method is the application of a technique for the determination of a specific analyte in a specific matrix.

17 **Procedure:** Written directions outlining how to analyze a sample.

PURPOSE OF CHEMICAL ANALYSIS

To characterize raw materials,

Asses the chemical safety of the product

Control the environment

ANALYSIS IN GENERAL CLASSIFIED INTO TWO TYPES

Nature of Information required

Size of sample material

BASED ON INFORMATION REQUIRED THE ANALYSIS IS DONE IN THE FOLLOWING WAYS

- Proximate Analysis
- Partial Analysis
- Trace analysis
- Complete analysis

THE LANGUAGE OF ANALYTICAL CHEMISTRY

Proximate Analysis Amount of each element is determined with no concern of sample. Ex. Proximate analysis of Coal which includes determination of Moisture, Volatile compounds, Ash content, fixed carbon

Partial Analysis It involves determination of selected constituent in the sample

Trace Analysis Trace analysis : It involves determination of specified components in the sample in a very minute quantity.

Complete Analysis It involves determination of proportion of each components in the sample .

ON THE SIZE OF THE SAMPLE USED

Macro analysis: Size of the sample ranges from more than 100 mg or more than 100uL

Semi micro analysis: Size of the sample ranges in between 10 mg- 100 mg or volume between 50-100 uL.

Micro analysis: Size of the sample ranges in between 1 mg- 10 mg or volume less than 50 uL.

METHODS OF ANALYSIS

Classical Methods of Analysis

Non Classical Methods of Analysis

ANALYTICAL METHODS

- Chemical methods
- Physical Methods
- Classical Methods
- Non Classical methods

ADVANTAGES OF CLASSICAL METHODS

- Simple procedure
- Cheaper equipments
- No specialized training required

CLASSIFICATION OF CHEMICAL METHODS OF ANALYSIS

Gravimetry
or
Gravimetric analysis

Volumetry
or
Volumetric analysis

CLASSIFICATION OF TITRIMETRIC ANALYSIS

- Acid Base Titration
- Precipitation Titrations
- Complexometric Titrations
- Redox Titration

NON CLASSICAL METHODS

CLASSIFICATION OF INSTRUMENTAL METHODS

- Optical methods
- Electroanlytical methods
- Separation methods
- Miscellaneous methods

ADVANTAGES OF NON -CLASSICAL METHODS

Fast
No tedious

Accurate and sensitive with greater precision

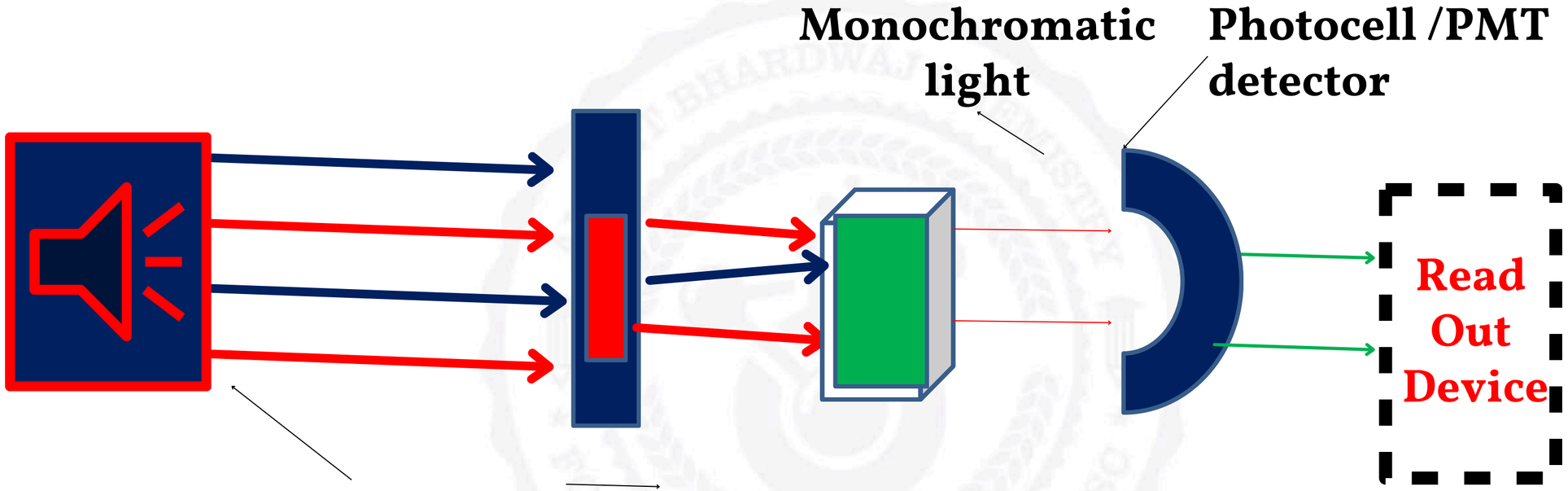
Small quantity of sample required

Ability to detect and estimate even trace quantity

OPTICAL METHODS

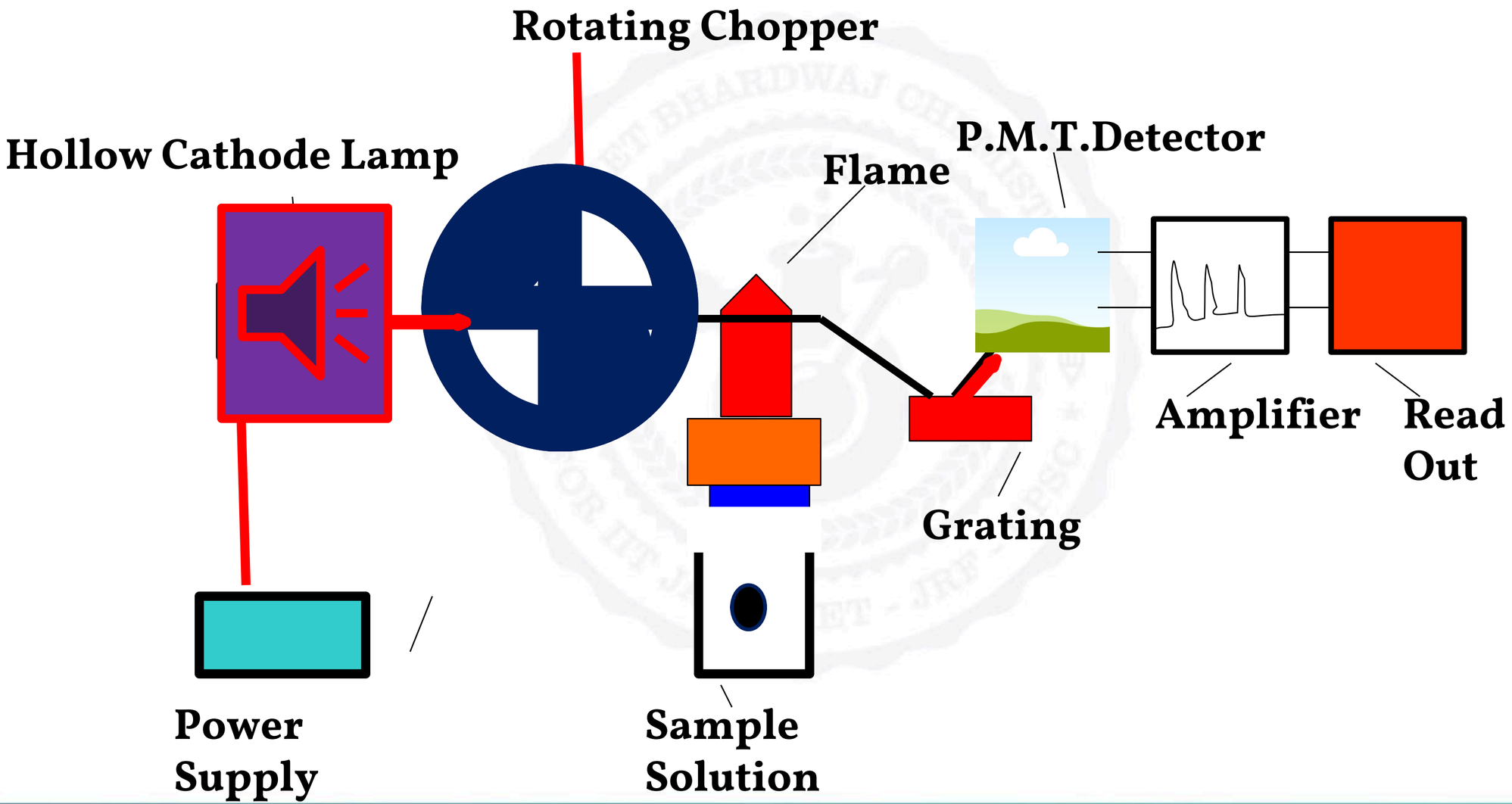
- Absorption spectroscopy
- Emission spectroscopy
- Atomic absorption spectroscopy
- Raman Spectroscopy
- Fluorophotometry
- Turbidimetry and Nephelometry
- Infrared Absorption Spectroscopy

2. INSTRUMENT: SINGLE BEAM COLORIMETER

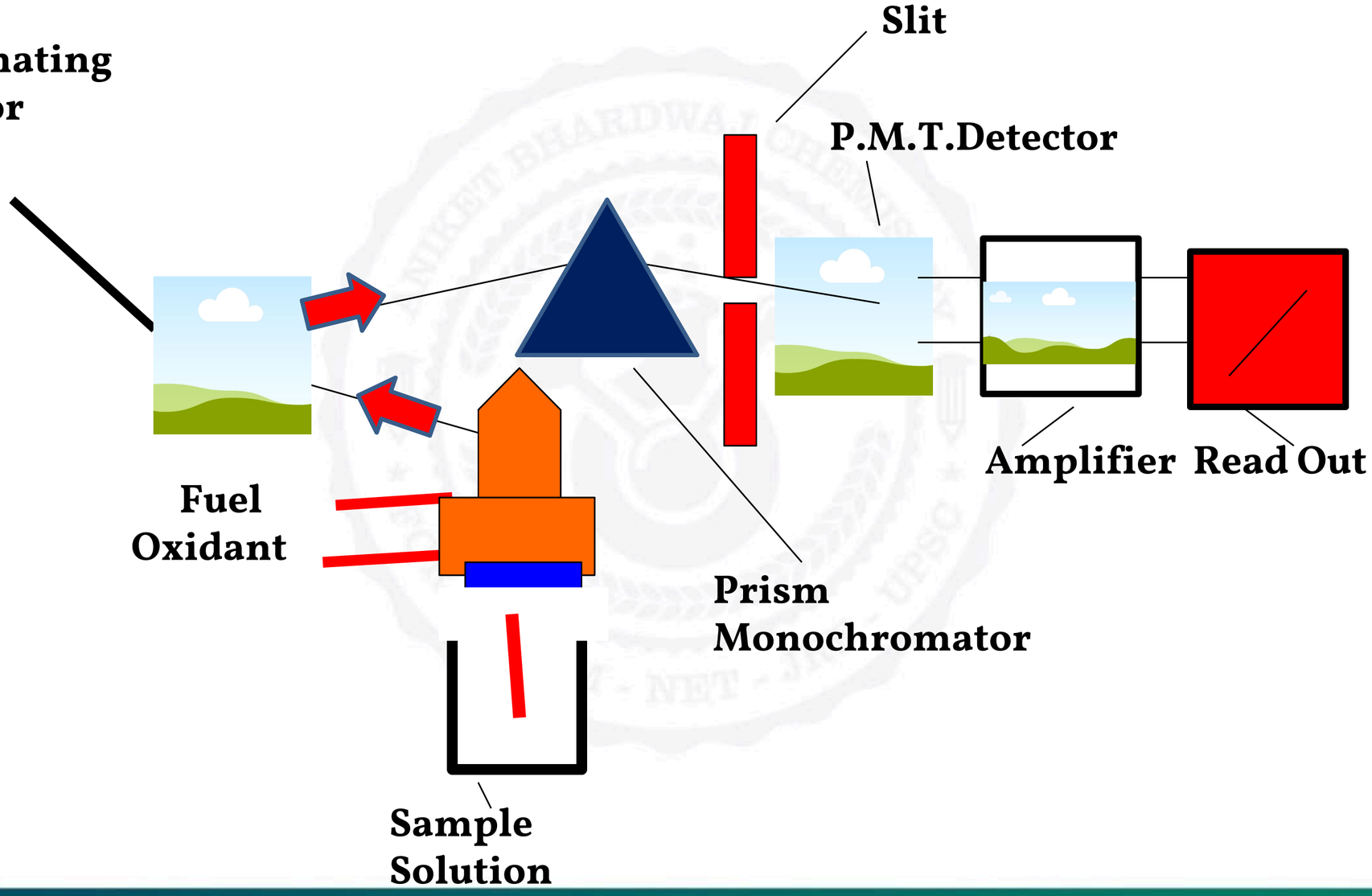


Absorption spectroscopy

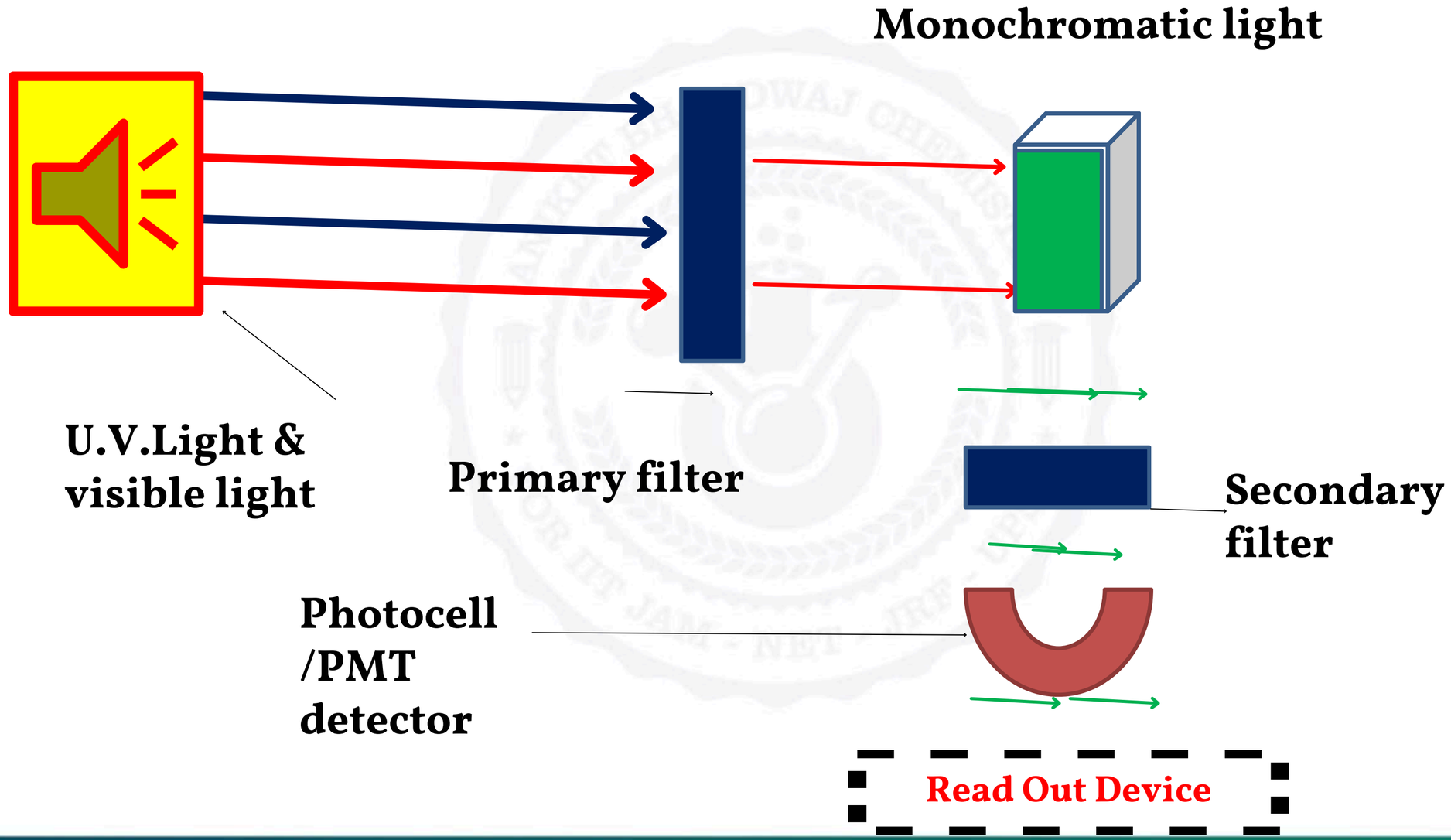
ATOMIC ABSORPTION SPECTROPHOTOMETER



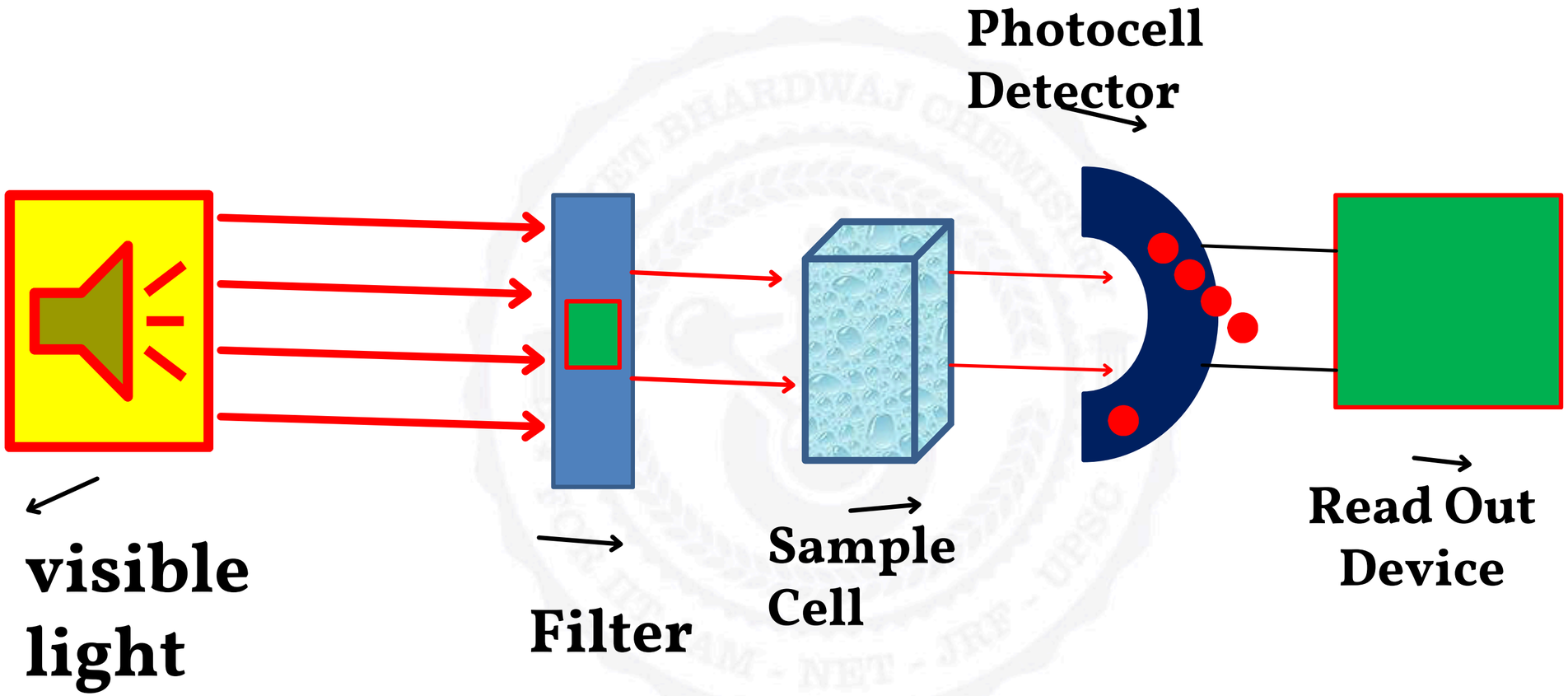
EMISSION SPECTROSCOPY- FLAME PHOTOMETER



2. INSTRUMENT: SINGLE BEAM FUORIMETER



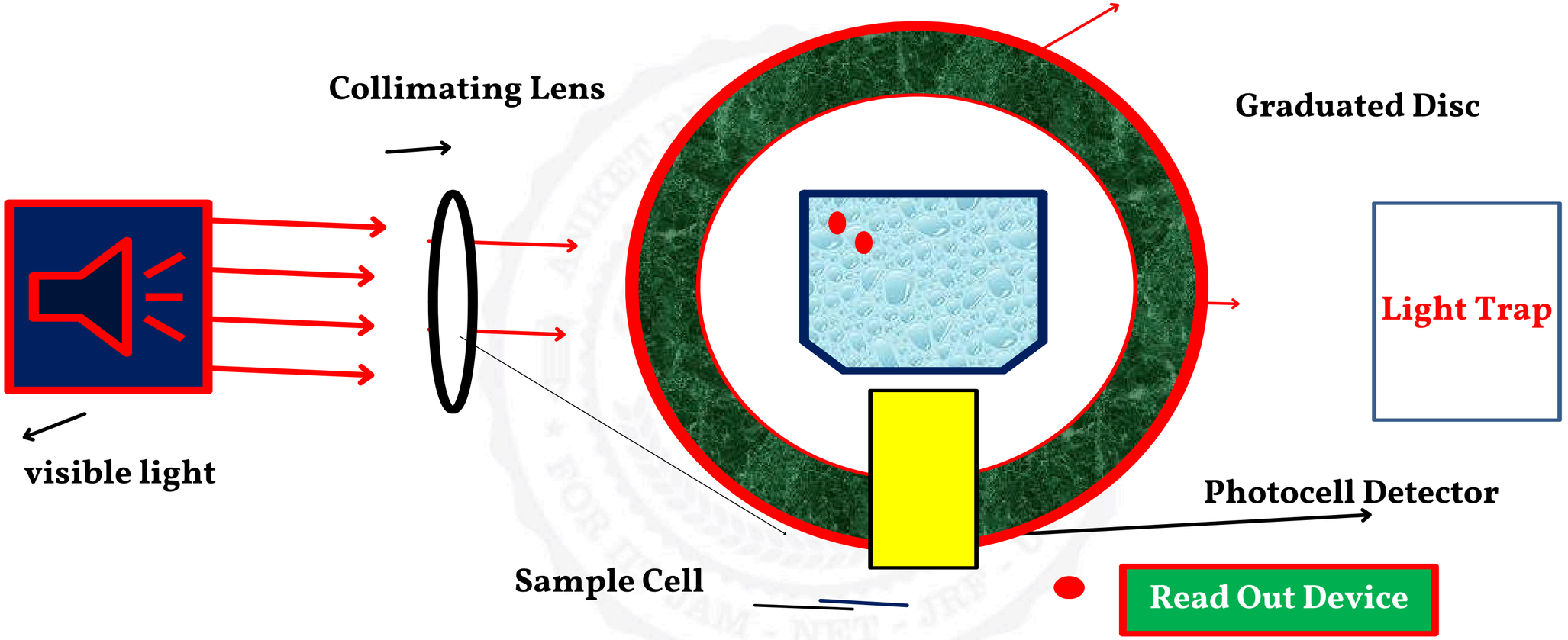
TURBIDIMETER



Technique is used when concentration of suspended particles are high

In this intensity of transmitted light is measured

NEPHELOMETER



Technique is used when concentration of suspended particles are less
In this intensity of scattered light is measured

ELECTROANALYTICAL METHODS OF ANALYSIS

- Potentiometry
- Amperometry
- Conductometry
- Polorography

RADIOANALYTICAL METHODS

Isotopic Dilution Methods

Neutron Activation Analysis

THERMAL METHODS

TGA
Thermogravimetric Analysis

DTA
Differential Thermal Analysis

SEPARATION METHOD

Classical Methods

Modern Methods

1.2 SAMPLING

Terms involved, importance of sampling, sampling techniques, sampling of gases, ambient and stack sampling, equipments used, sampling of homogenous and heterogeneous liquids, sampling of static and flowing liquids, methods and equipments used, sampling of solids, importance of particle size, and sample size, samples used In order to understand the criteria for evaluating the utility of the analytical techniques, need for the reduction in sample size, methods of reduction in sample size, collection, preservation and dissolution of the sample.

TERMS INVOLVED IN SAMPLING

- The Population or Universe
- Sampling Techniques or Procedures
- Sampling Unit
- Increment
- Gross Sample
- Sub sample
- Analysis Sample

1. The Population or Universe: The bulk material from which sample is to be drawn is termed as Population or Universe.

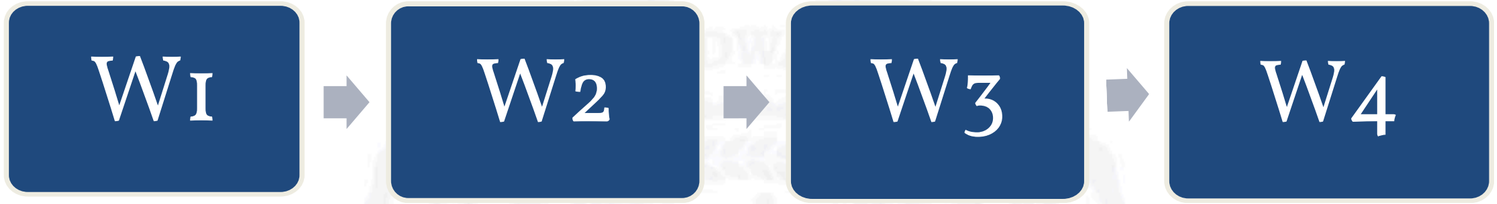
2. Sample: A small portion of the universe drawn for the purpose of analysis and which posses all essential characteristics of the universe is called as sample.

3. Sampling Techniques or Procedures: The series of steps that is to be carried out to obtain a sample Sampling Technique or sampling procedure.

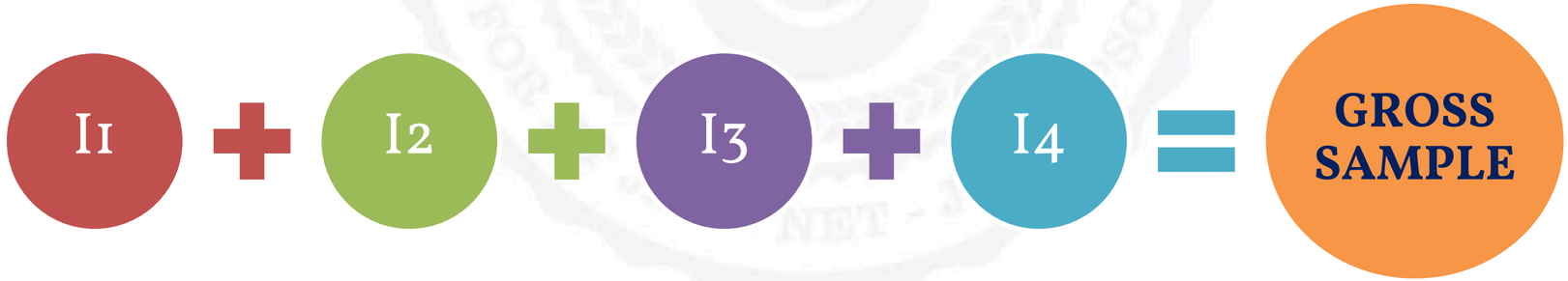
4. Sampling Unit: The minimum size package in the consignment which sample may represent is known as sampling unit.

5. Increment: A stated amount of the material that is withdrawn from the sampling unit is defined as Increment.

Sampling Unit: The minimum size package in the consignment which sample may represent is known as sampling unit.



Increment: A stated amount of the material that is withdrawn from the sampling unit is defined as Increment.



Gross Sample: The total sample obtained by mixing or blending all increments is known as gross sample.

Sub sample: A smaller size sample produced by subdivision of the gross sample and which posses all essential characteristics of the gross sample is known as sub-sample.

Analysis Sample: An accurately weighed amount of sub sample taken actually for analysis is known as analysis sample.

PURPOSE OF SAMPLING

- Judging acceptability
- Detecting Contamination
- Identification of Material

JUDGING ACCEPTABILITY

To know the material from which sample is withdrawn meets the essential requirements such as purchase or sales specification so that material can accepted or rejected.

For this purpose sample should represent the whole quantity under consideration

DETECTING CONTAMINATION

The second Objective or purpose of sampling is to ensure that material is free from contamination.

For this purpose sampling is carried out such that sample will give maximum assurance of finding the contamination.

IDENTIFICATION OF MATERIAL

Third purpose of sampling is to identify the material. A carefully drawn the sample can accurately give information of the material.

TYPES OF SAMPLING

Random Sampling

Non-Random Sampling

RANDOM SAMPLING

Random sampling involves the selection of material without any bias or prejudice.

In this method every part of bulk material has an equal chance of being picked up as a sample.

This technique of sampling requires a minimum knowledge of the universe under consideration. If the material is homogenous it is easy to sampling.

If the material is homogenous, random sampling is easy to perform.

If the material is heterogeneous sampling becomes time consuming.

In random sampling bulk material is divided into groups. division of groups is done on the basis of similarity in characteristics. from each groups sample is withdrawn at random.

NON-RANDOM SAMPLING

Sampling is carried out in more scientific way than random sampling.

In this sampling better sample is not obtain than random one.

Ex. in pharmaceutical industry sampling of tablet is carried out systematically. After every hundred tablet one tablet is pick up for analysis..

DIFFICULTIES ENCOUNTERED IN SAMPLING

- Lack of prior information:
- Physical Nature: Ex. In pile of coal, the interior portion is not easily available Or sampling of rock material is difficult.
- Excessive cost: Time, labour, and money. Ex.if sampling units are too large, sampling cost will go up.

Sampling of liquids
Care required for sampling of liquids

1	2	3
Cleanliness of apparatus and containers	Preservation of sample composition	Positive identification of samples

CLEANLINESS OF APPARATUS AND CONTAINERS

1. Sample containers should be washed repeatedly with tap water and then distilled water.
2. Containers should be dried under sunlight or by warm air.
3. Clean hands should be used while collecting sample
4. Gloves should not be worn except when necessary ,if used it should be clean.
5. Containers should be labeled properly after sampling; date, location, time, purpose of sampling ,nature of sample etc.

PRESERVATION OF SAMPLE COMPOSITION

- If the sample contains solid particles or droplets of immiscible liquid, care must be taken such that all particles should transfer to sample containers.
- Sample should not allow to solidify.
- Dissolved and entrained gases should not allowed to escape.
- Entraining air in the out side should be avoided sample.
- During handling and transportation sample should be protected against breakage, evaporation, leakage, exposure to sunlight and entry of dust and air.

POSITIVE IDENTIFICATION OF SAMPLES

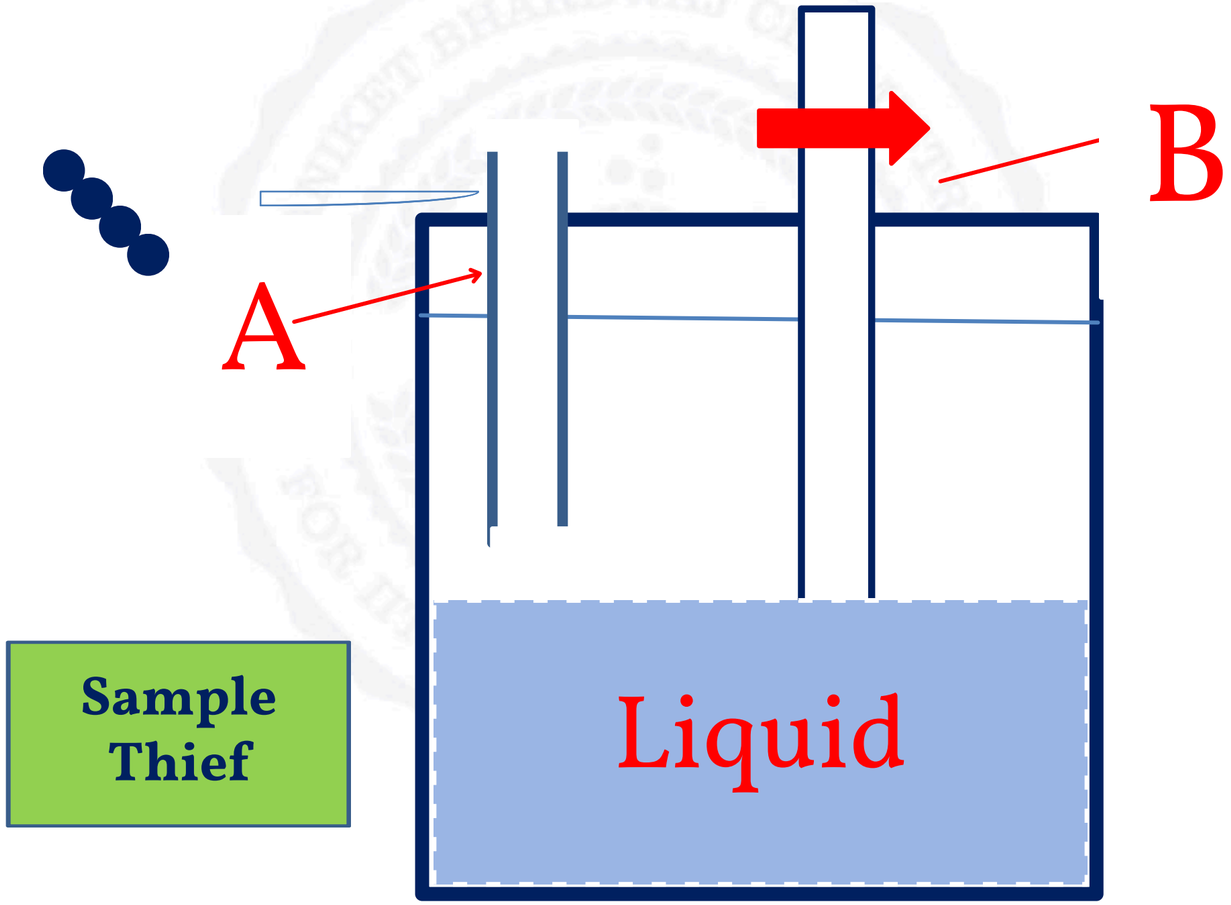
1. Proper location for sampling should be identified.

2. Consideration of location for sampling must be justified.

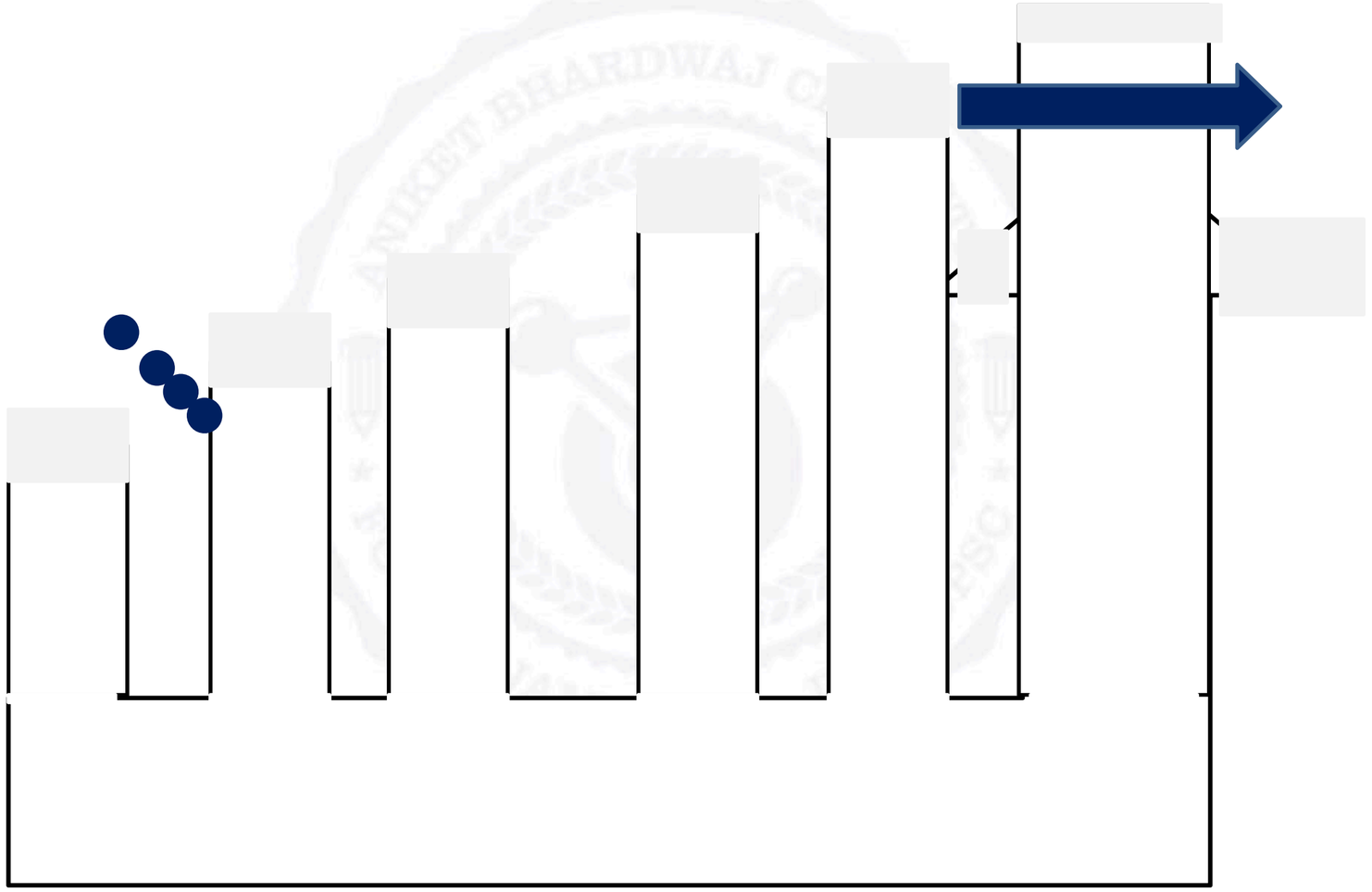
TYPES OF LIQUIDS TO BE SAMPLED

- Homogenous Liquids
- Heterogeneous liquids or immiscible liquids
- Liquids containing Emulsions or unstable suspension
- sampling of liquids containing partially crystallized solids
- Sampling of static and flowing liquids.
- Sampling of liquids from various containers.

APPARATUS FOR SAMPLING OF HOMOGENOUS LIQUIDS HETEROGENEOUS LIQUIDS OR IMMISCIBLE LIQUIDS

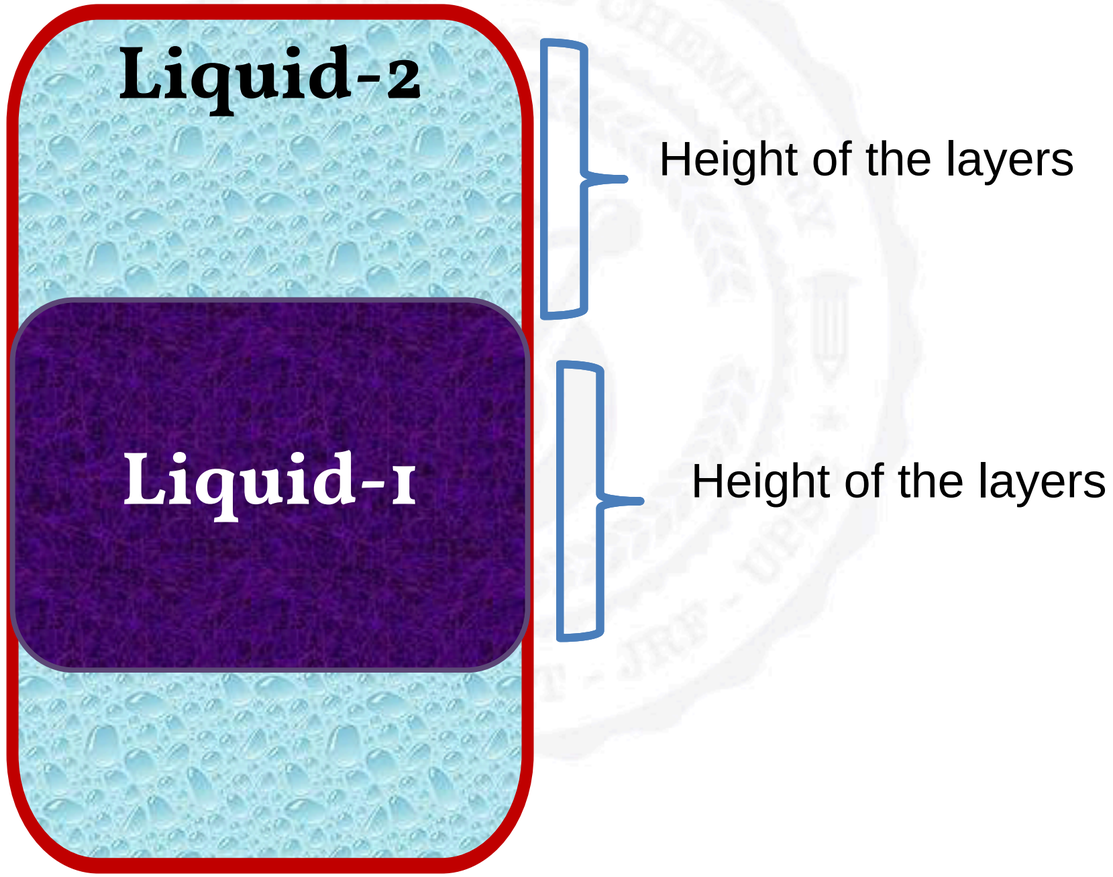


DEVICE FOR SAMPLING OF STATIC AND FLOWING LIQUID



HETEROGENEOUS LIQUIDS OR IMMISCIBLE LIQUIDS

1) Two immiscible liquids separating into two layers :



Sample thief is used to with draw the sample so that gross sample can be prepared.

HETEROGENEOUS LIQUIDS OR IMMISCIBLE LIQUIDS

2) Emulsion or unstable suspension:

- Two phases are separated by filtration ,
- Solid and liquid are separated
- The two phases are weighed and then sampled separately.

If above is not feasible the entire suspension is stirred vigorously and resulting liquid is sampled.

Sample thief is used to with draw the sample so that gross sample can be prepared.

HETEROGENEOUS LIQUIDS OR IMMISCIBLE LIQUIDS

3) sampling of liquids containing partially crystallized solids :

- Semi solidified liquid or liquid containing crystallized solids are heated,
- Heating is continued till solid dissolves or melts into liquid.

Then sample is withdrawn by sample thief.

SAMPLING OF GASES

- Gases from gas pipe line
- Gas from gas well
- Processing plants
- Storage holders such as tank
- Open atmospheres

DEVICES FOR SAMPLING OF GASES

Sampling probe: Sampling line attached to container or vessel.
It is injected into gas containers/pipe/well etc.

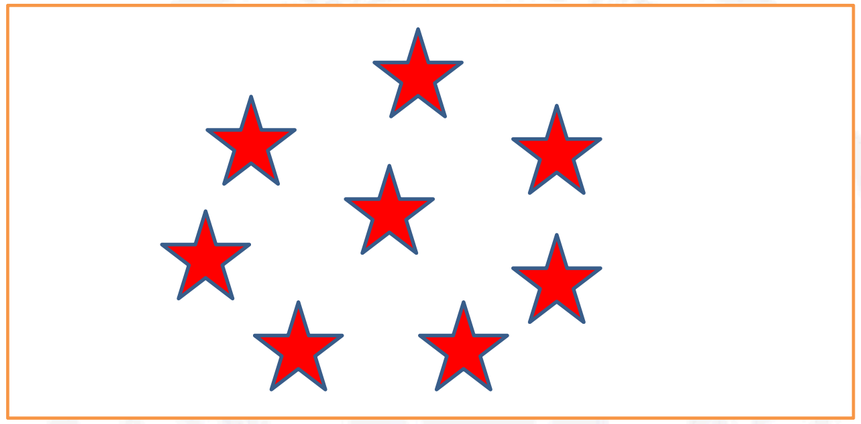
Sample Container: Vessel in which gas sample is collected; Size of containers may vary from 250 cm3 to several cubic meters.

GENERAL PROCEDURES FOR SAMPLING OF GASES

- Sample containers to hold the gas should be of glass
- The containers are connected to stopcock at both ends which will facilitate easy flushing.
- The joints are made from glass to make container leak proof.
- Rubber tubing should be avoided to avoid the reaction.
- The stopcock are carefully cleaned and then lubricated every time before every time.
- The analysis of gas sample should be carried out simultaneously or immediately, after the sampling .
- Flushing, displacement by liquid and expansion into evacuated vessel are general methods used for sampling of gases.
- In flushing method, the sample container should be flushed with the gas to be sampled ten to fifteen times.

AMBIENT SAMPLING

Ambient sampling : Sampling of atmospheric gases is called as Ambient sampling



The definite area is selected for sampling.

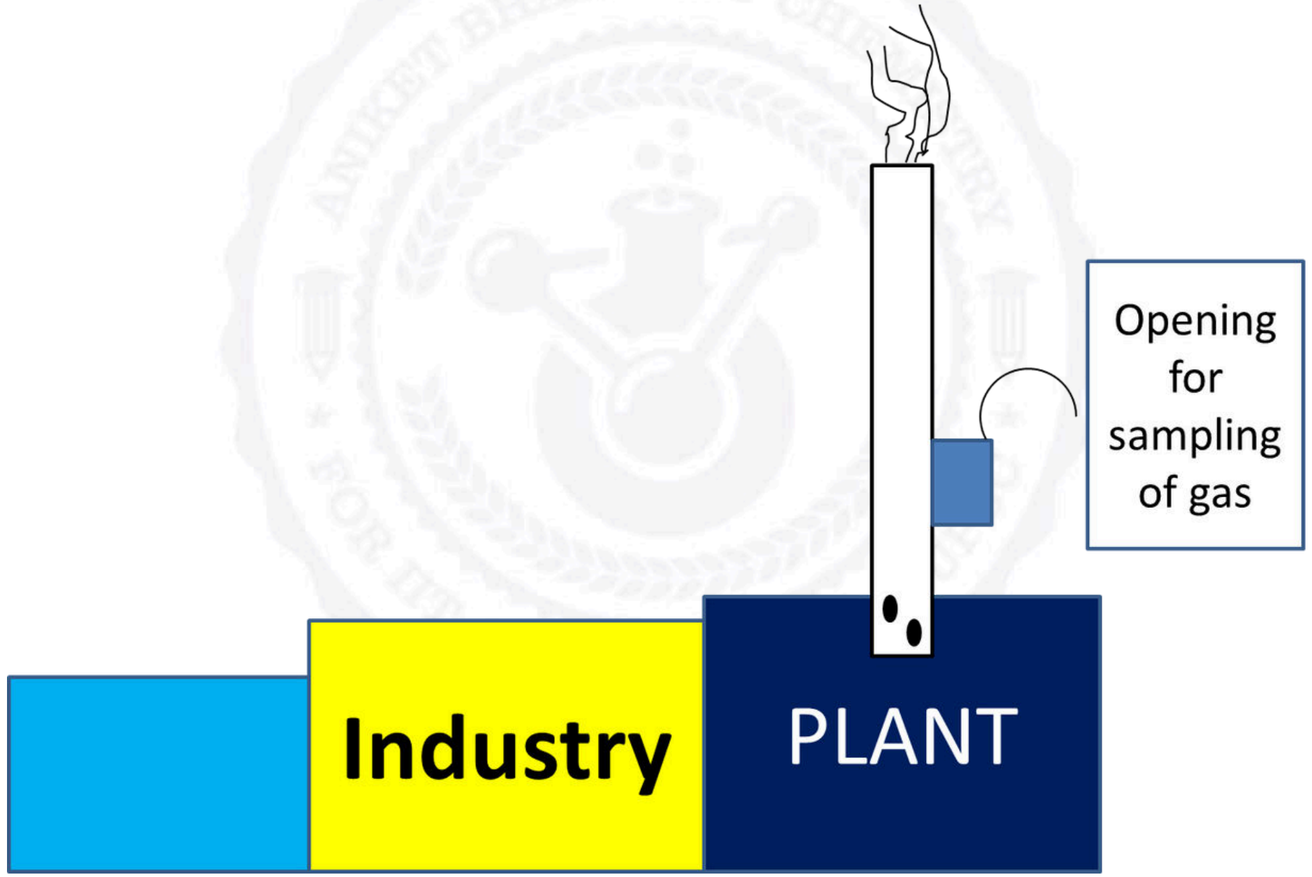
Sampling stations are decided on the basis of grid pattern.

Sampling of atmospheric gases should be carried out region wise and area wise and time wise.

For. Ex. Sampling of atmospheric air in Thane city

STACK SAMPLING

Sampling of gases released from industry is called as stack sampling.



STACK SAMPLING

Industrial gases are generally sampled continuously.

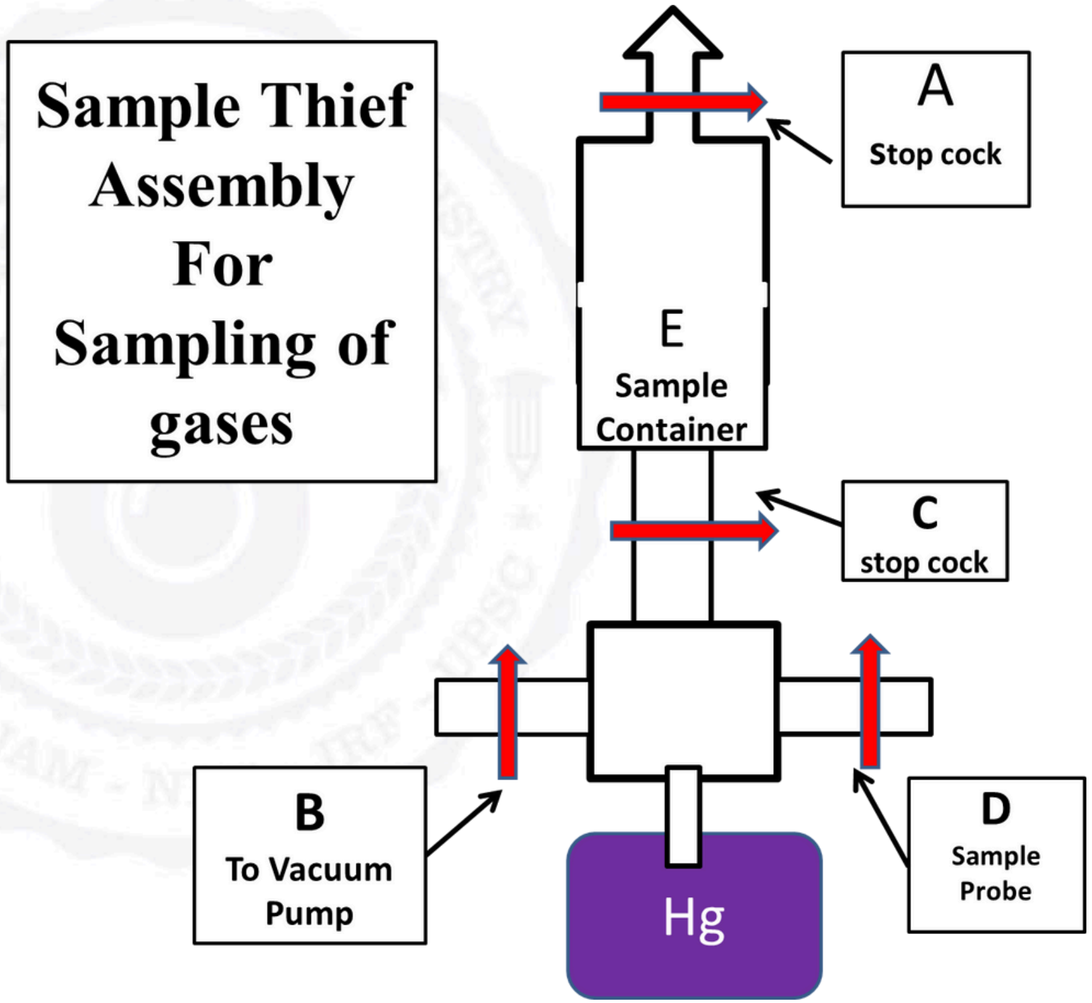
While sampling it should be ensured that sample collected should represent constant fraction of total flow of all portions of streams are sampled.

SAMPLING OF GASES

Two methods are used

1) Static method

2) Dynamic method



STATIC METHOD OR EVACUATION METHOD

Sample container is evacuated by means of vacuum pump.

- For evacuation stopcocks B&C are kept open and stopcocks A &D are kept closed.
- After evacuation the container is also warmed to remove adsorbed gases on wall of the container. Then stopcock A and B are closed ,C and D are open. The gas to be sampled is allowed to entered into sample container through sample probe D
- The excess of gas escape and immersed in a pool of mercury.
- The evacuation and filling is carried out repeatedly to get desired sample.
- Use: This method is useful when small amount of gas is available

DYNAMIC METHOD

Sample container is evacuated by means of vacuum pump.

During vaccumisation stop cock C is closed and A ,E AND F are kept open.

During sampling, gas from the cylinder are admitted into sample container.

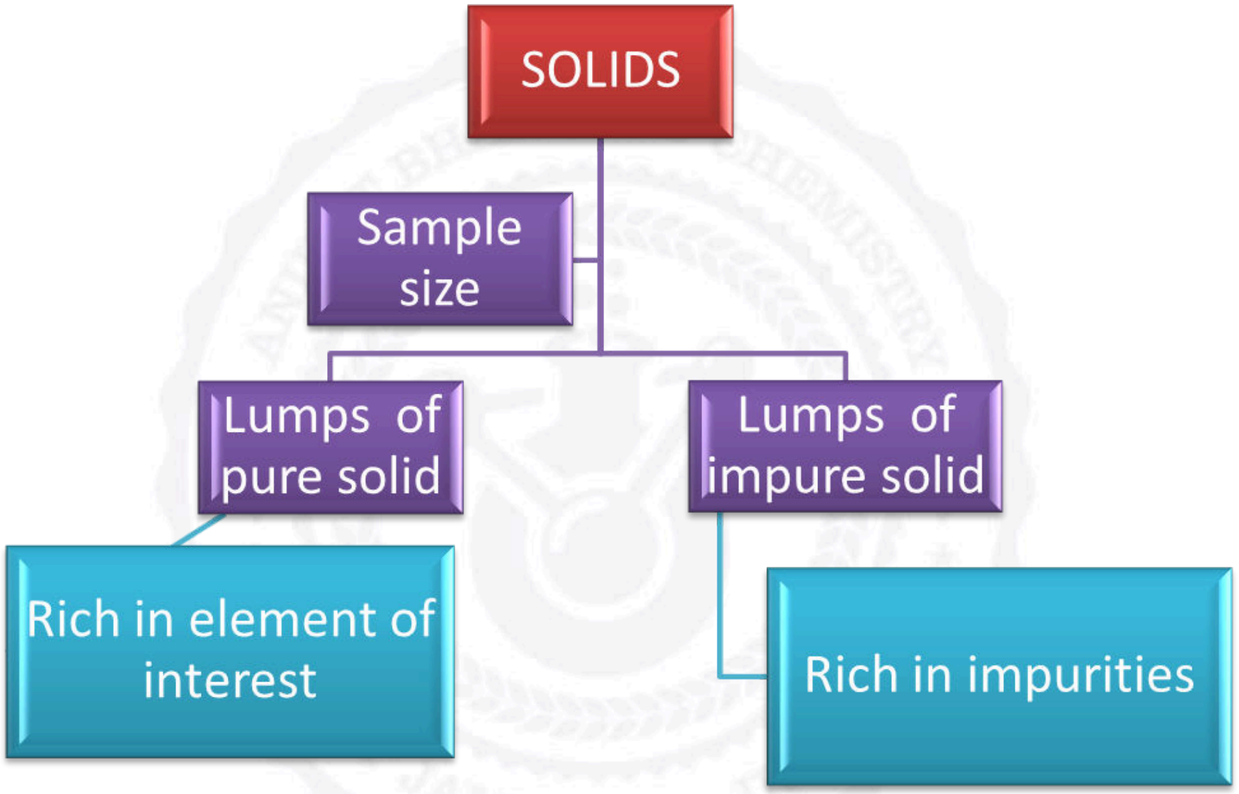
By repeating procedure several times, the container is flushed to remove the residual gas and desired sample will be obtained.

Use: This method is useful when large amount of gas is available

SAMPLING OF SOLIDS

- As solid are heterogeneous it is difficult to carry out sampling of solids.Ex.in ores certain lumps are rich in the element of interest and may rich in impurities such as silicates.Lumps of pure solid and lumps of impurities in different sizes may lead to errors in sampling. Such errors can reduced by crushing larger lumps into smaller size or by taking large quantity of the sample.
- In this context the concept of bulk ratio is important.
- BULK RATIO= Weight Of The Sample \Total Weight Of Bulk Material
- SIZE TO WEIGHT RATIO= weight of the largest particle/weight of the sample

HETEROGENEOUS MATERIAL



Such errors can reduced by crushing larger lumps into smaller size or by taking large quantity of the sample.

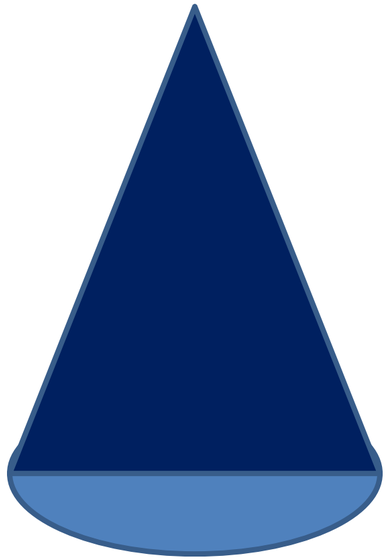
- In this context the concept of bulk ratio is important.
- BULK RATIO= Weight Of The Sample / Total Weight Of Bulk Material
- SIZE TO WEIGHT RATIO = weight of the largest particle/weight of the sample.
- This concept is applied to determine the number and size of increment.

For sample which is true representative of bulk material bulk ratio of the sample is large and size to weight ratio should be small

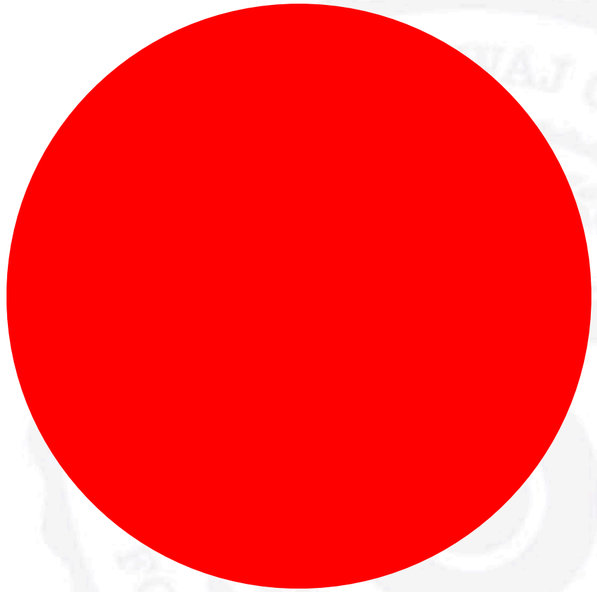
SIZE REDUCTION

- Coning and quartering
- Long pile and alternate shovel method
- Method using riffles

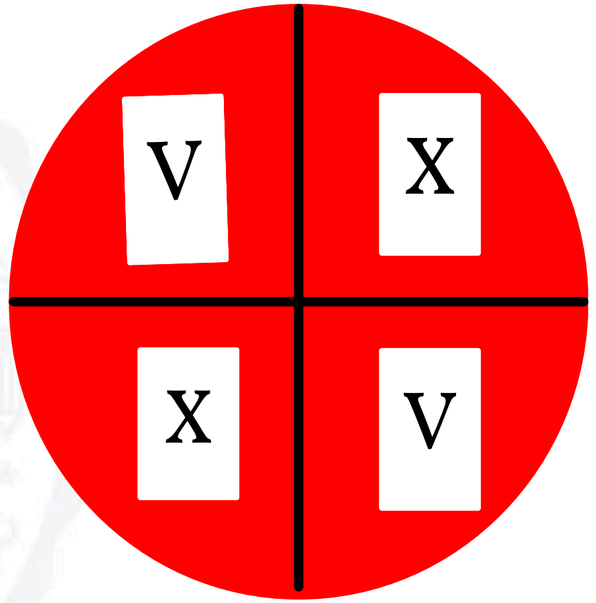
CONING AND QUARTERING



Cone of the material

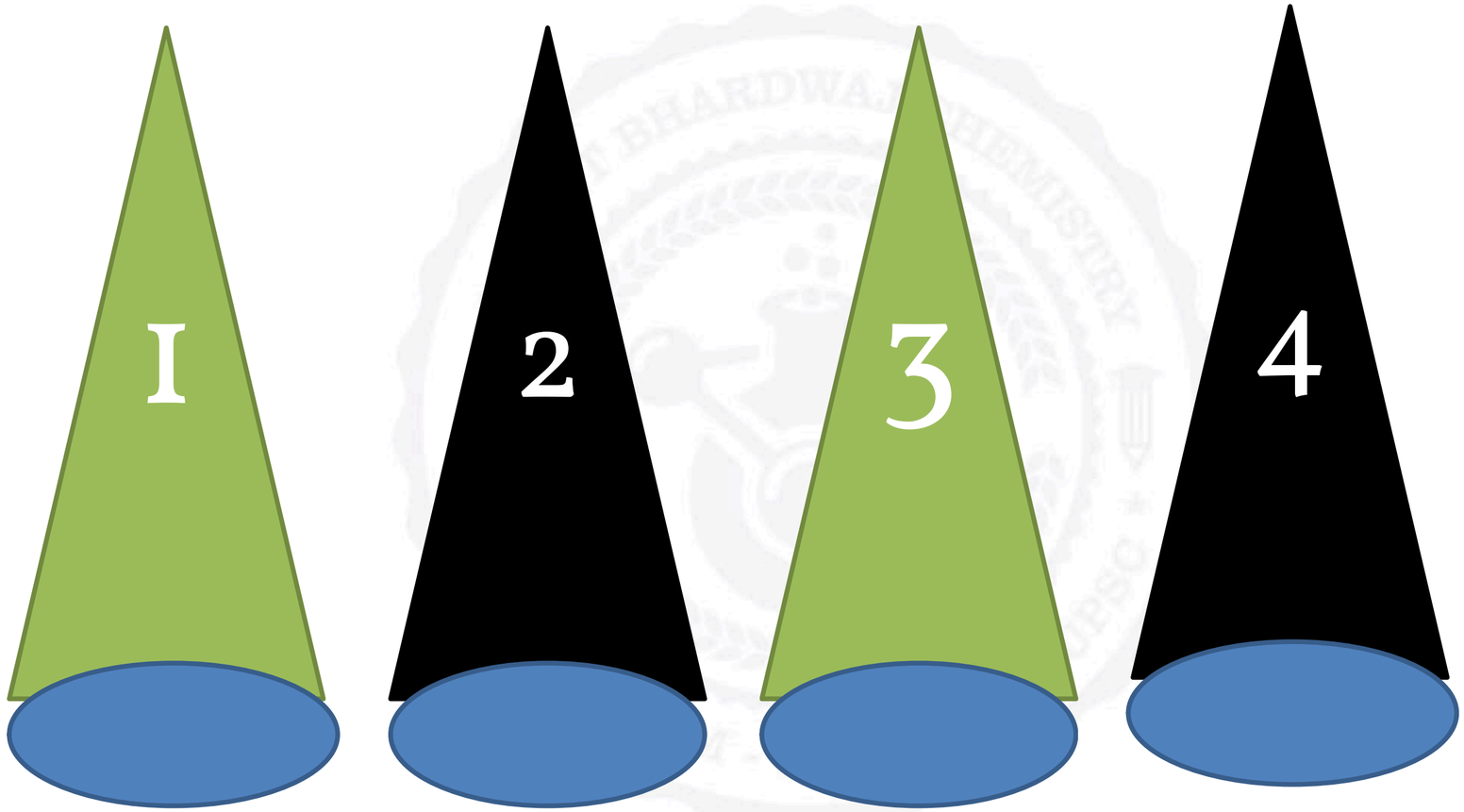


Material flattened to circle



Two opposite parts selected other two are rejected

LONG PILE AND ALTERNATE SHOVEL METHOD



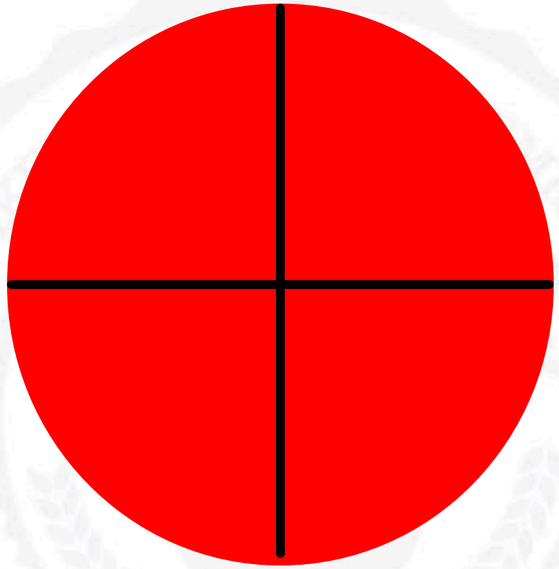
1 and 3 are selected and 2 and 4 are rejected or vise versa

ROLLING AND QUARTERING

V X

X V

Two opposite parts selected other two are rejected



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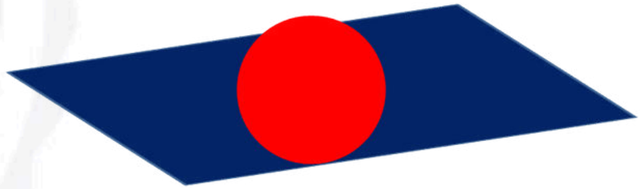
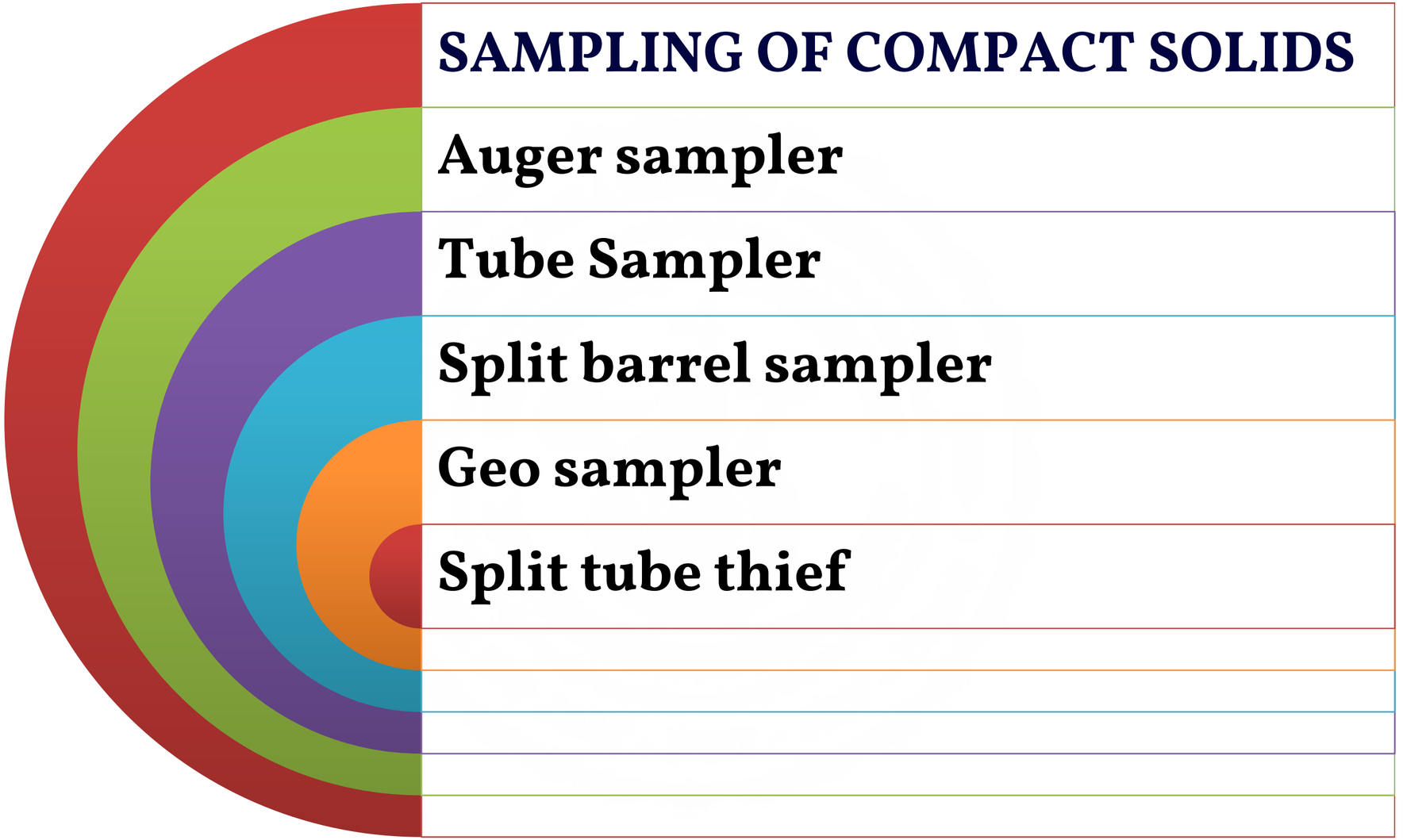
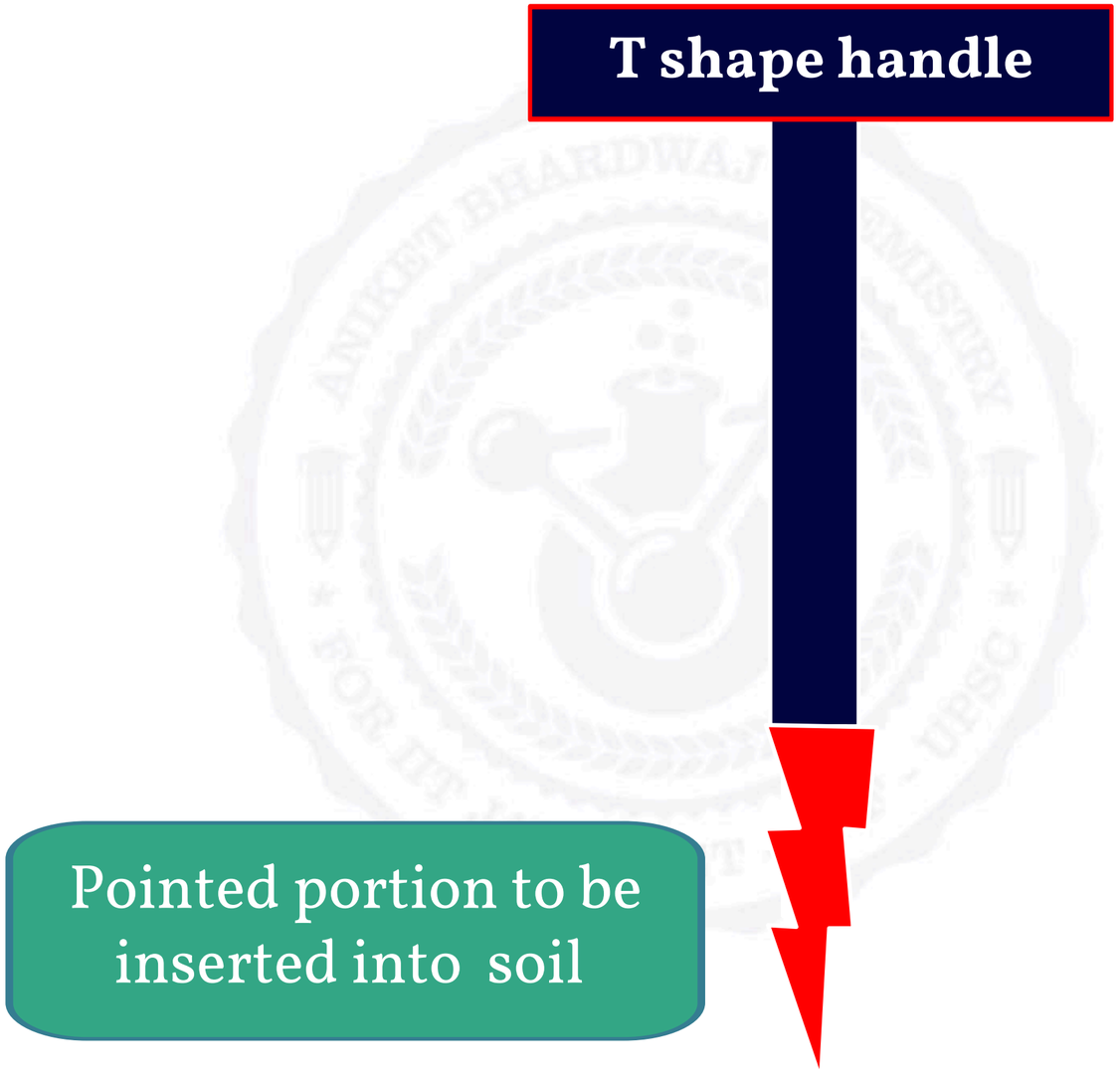




SAMPLING OF COMPACT SOLIDS

- Auger sampler
- Tube Sampler
- Split barrel sampler
- Geo sampler
- Split tube thief

AUGER SAMPLER



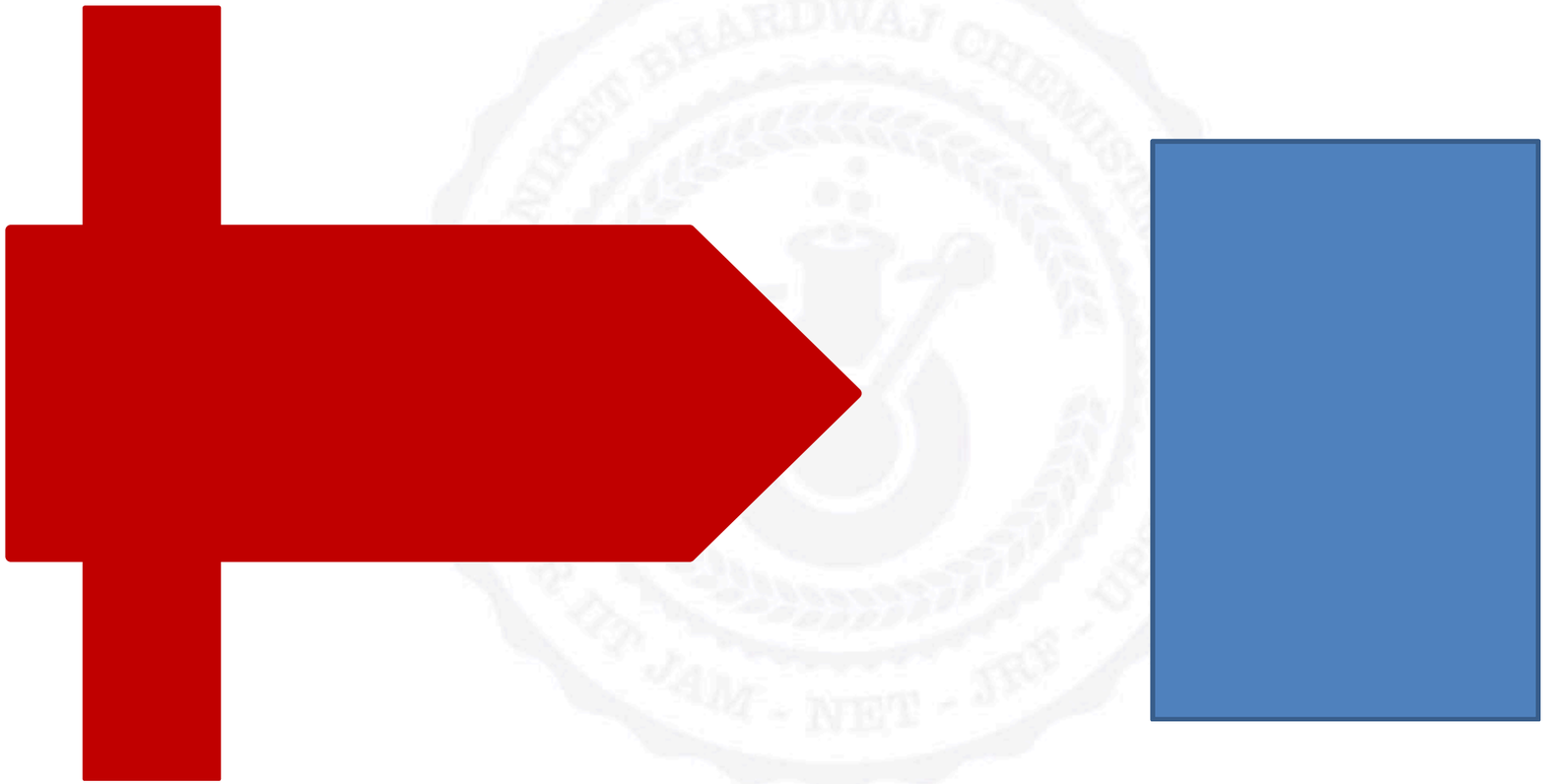
Tube Sampler: It is thin walled tube ,5 to 10 cm in diameter. It is used for penetration of predrilled hole.

Split barrel sampler: This is drilling equipment attached to driller. The driving head made from hard steel. It is 45 cm long. it is detached from the coupler and opened to remove sample .

Geo sampler: It is for collection of sample from planetary rock.

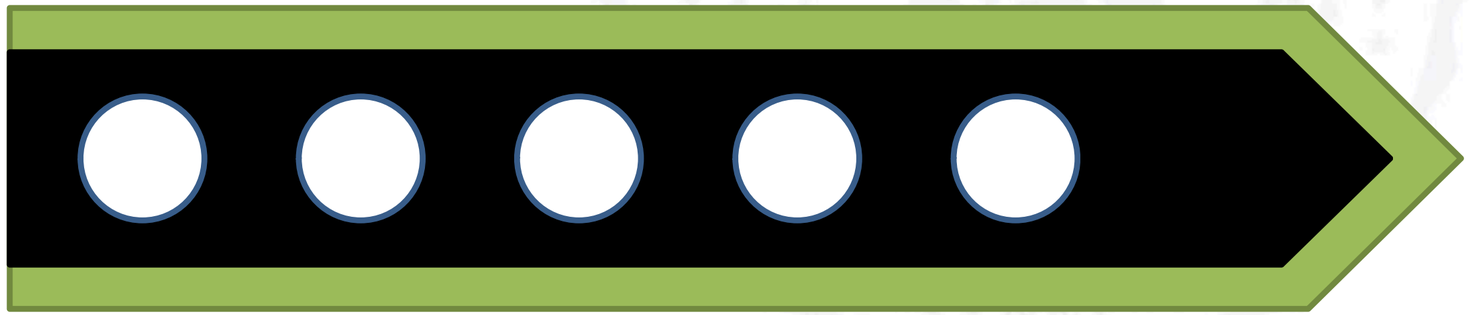
Split tube thief: A slot running through the entire length of the tube. The end of the tube is very sharp. The sharp end can pierced into gunny bag.

SPLIT TUBE THIEF



SAMPLING OF PARTICULATE SOLIDS

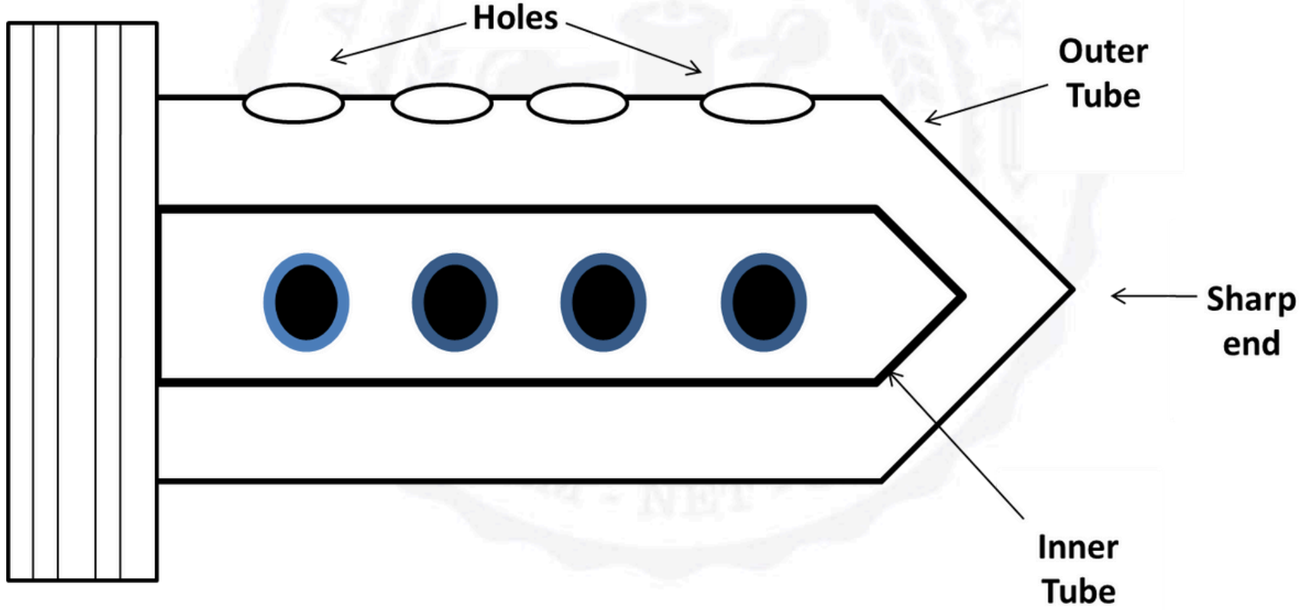
- Sampling of particulate solids: dry and free flowing Material called as particulate solids.
- Concentric tube thief



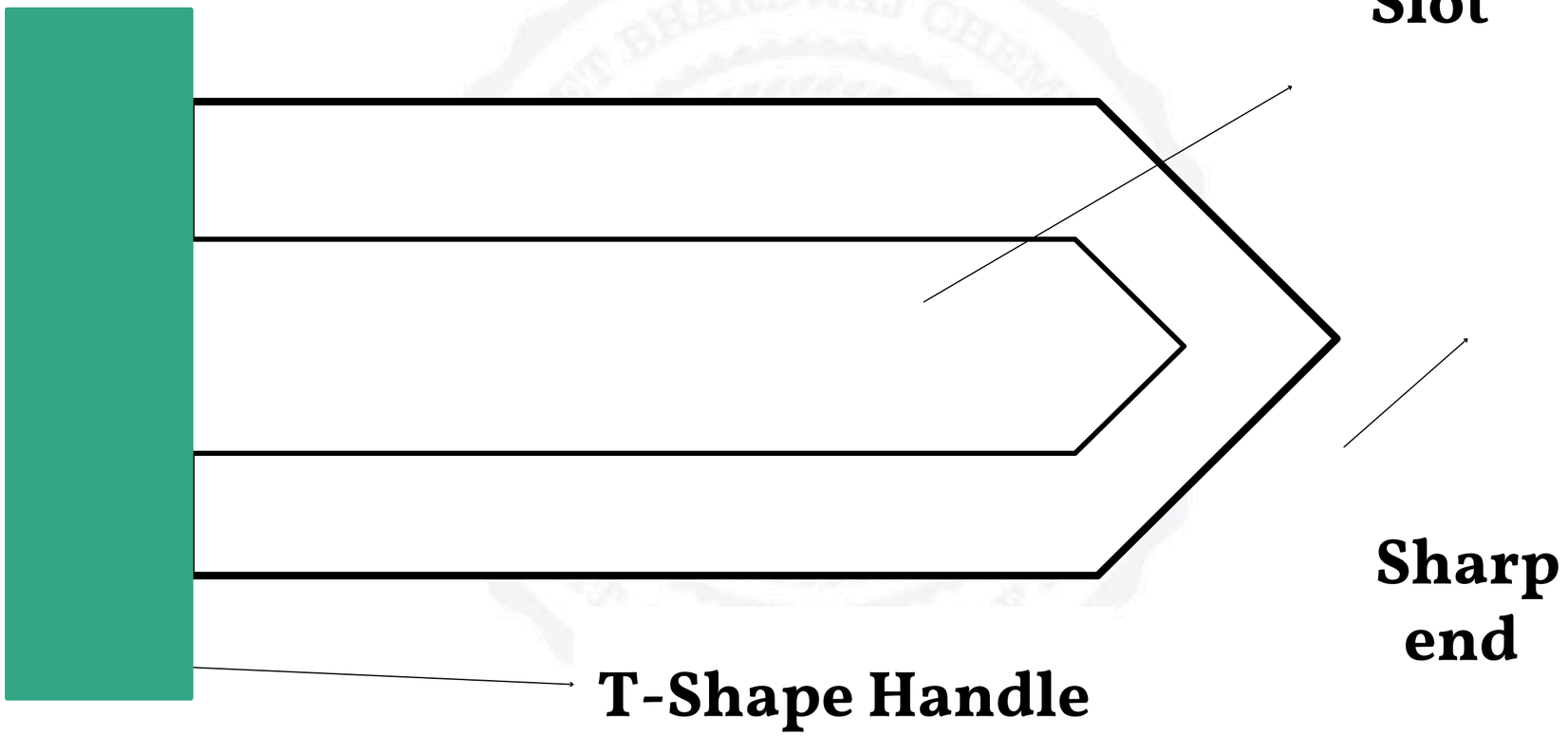
Solid material

CONCENTRIC TUBE THIEF

- Sampling of particulate solids: dry and free flowing Material called as particulate solids.
- Concentric tube thief



SPLIT TUBE THIEF



HAND SCOOP

COLLECTION ,PRESERVATION AND DISSOLUTION OF SAMPLE

- COLLECTION: Collection of sample depends on type of sample ,its physical state, and its chemical state.

- When there is gap between collection and analysis preservation of sample is necessary.

PRESERVATION OF SAMPLE

- When the sample is preserved its physical and chemical composition should remain same.
- Sample should not react with the environment nor with the sample container.
- Component of the sample should not react among themselves.
- Composition of sample should not change due to physical processes ex.volatilisation

DISSOLUTION OF THE SAMPLE

Most analyses are performed on solutions of the sample. Therefore, suitable solvent is required to dissolve the sample rapidly and under conditions in which there is no loss of the analyte. The dissolution process depends on the nature of the sample material.

TWO MOST COMMON METHODS EMPLOYED IN DISSOLVING INORGANIC SAMPLE ARE :

(1) Treatment with Acids : hydrochloric acid, nitric acid, mixture of hydrochloric and nitric acids, sulphuric acid or perchloric acid, and

(2) Decomposition by Fluxes: fusion with an acidic or basic flux followed by treatment with water or an acid. Organic solvents are preferentially taken to dissolve the samples of organic nature. However, special methods are to be developed to dissolve a silicate material, a high molecular weight polymer or a specimen of animal tissue.

FLUX	Crucible
Na_2CO_3	Pt.
Na_2CO_3 + KNO_3 Na_2O_2	Pt.

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