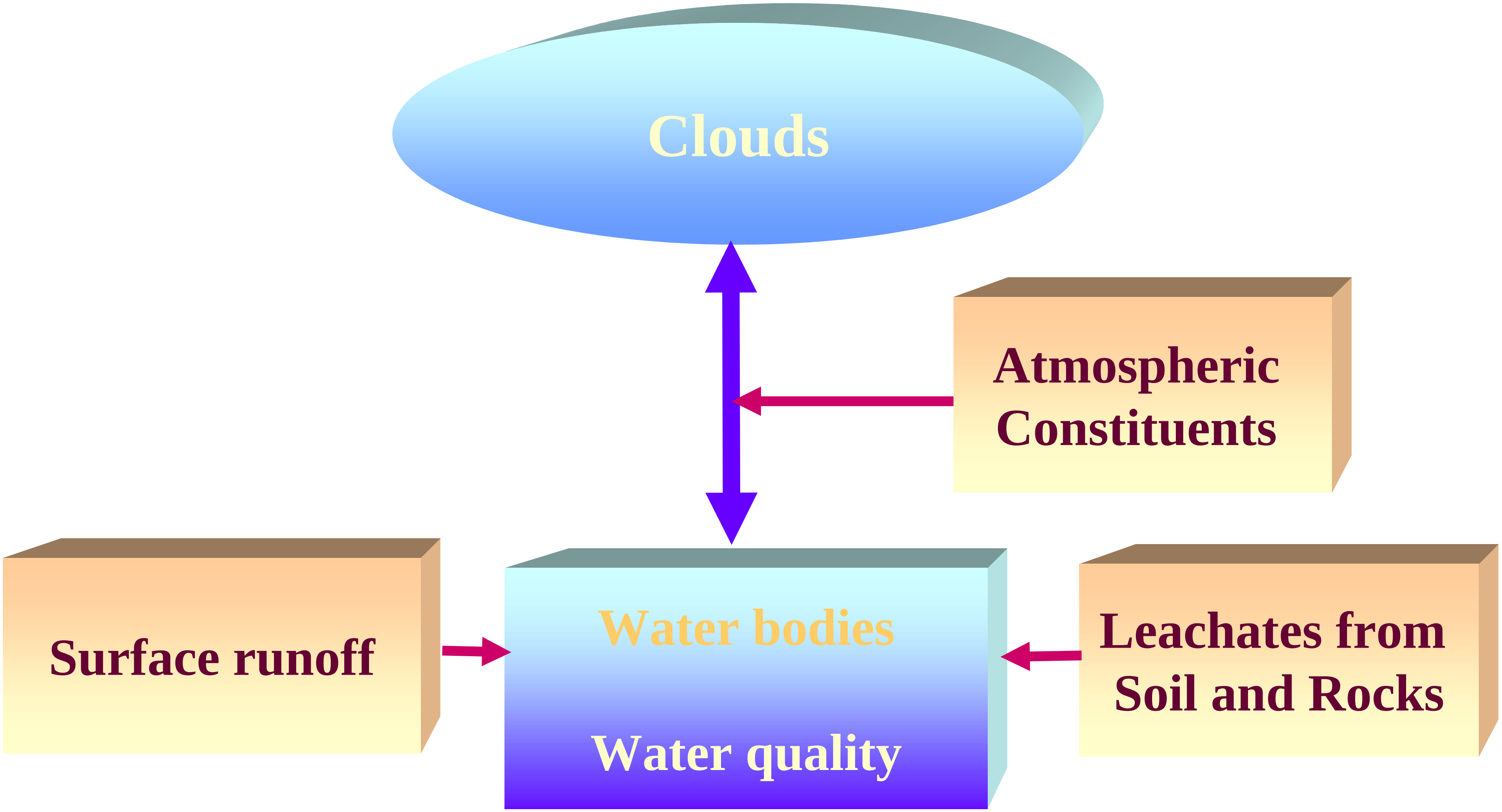
Water Quality Analysis
Laboratory Methods

Dr. (Mrs.) Leena Deshpande

National Environmental Engineering Research Institute (NEERI), Nagpur
Council of Scientific & Industrial Research,
New Delhi, Govt. of India

Dispersion & Dissolution in Water



Clouds

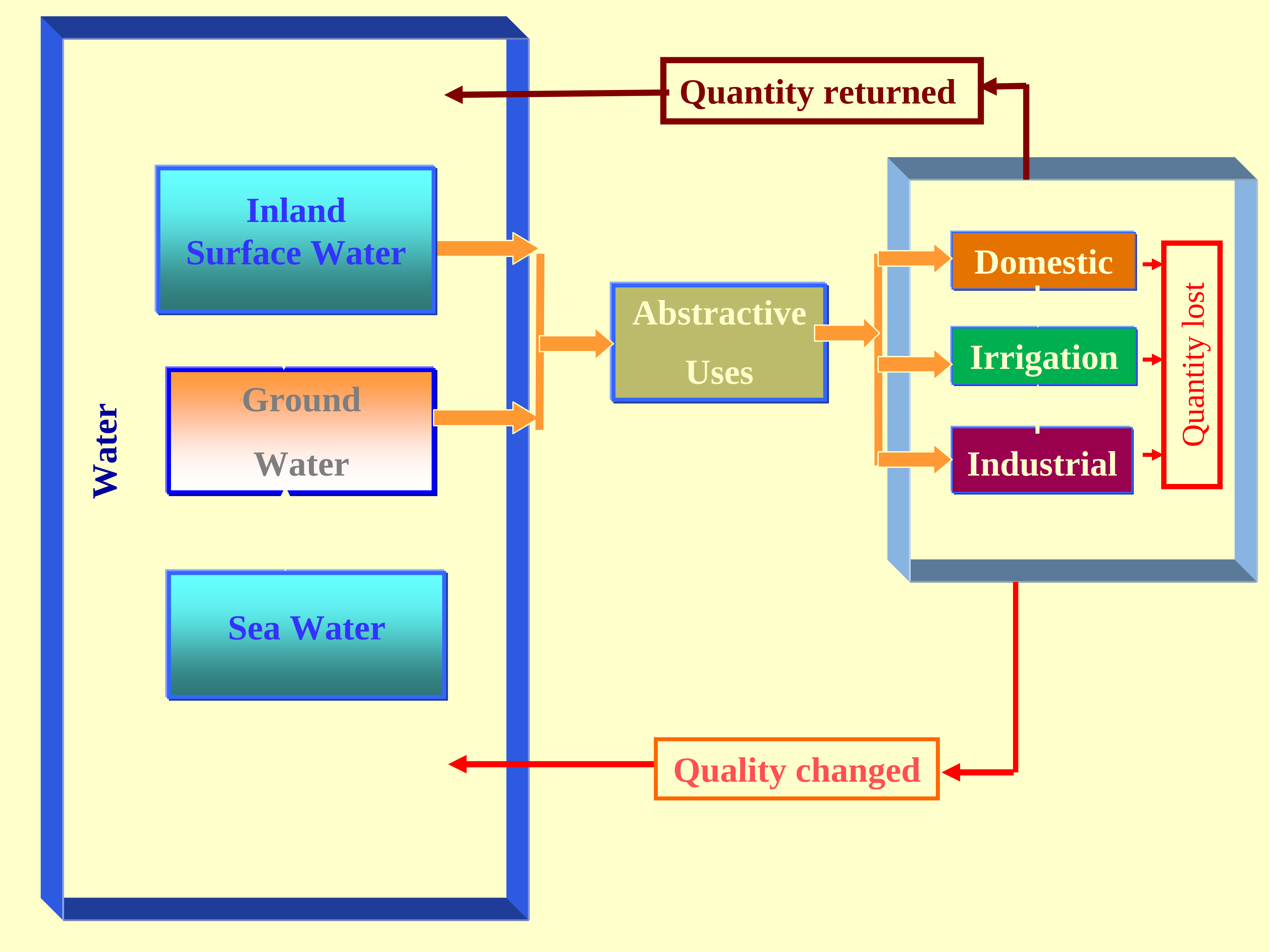
Atmospheric Constituents

Surface runoff

Water bodies

Water quality

Leachates from Soil and Rocks



Water

Inland Surface Water

Ground Water

Sea Water

Abstractive Uses

Domestic

Irrigation

Industrial

Quantity lost

Quantity returned

Quality changed

Water

Non-abstractive uses

- Transport
  - Sea
  - In-land Waters
- Food
  - Fishing
  - Sea Food
  - Pond Food
  - Lean Period River bed crops
- Recreation
  - Swimming
  - Sunbath & Swimming
  - Picnic Spot
  - Water Sports
  - Pilgrimage
- Eco-system
  - Conservation
  - Rejunvetion
  - Technical interferences
  - Technological Interventions

Adverse Effects of Impurities

Problems

- Aesthetically not acceptable and Palatability decreases
- Health related problems
  - affect mucous membrane
  - gastro-intestinal irritation
  - Dental and skeletal fluorosis
  - Methaemoglobinemia
- Encrustation in water supply structure
- Adverse effects on domestic use

Constituents Responsible

- Clay, Silt, Humus, Colour
- pH

- Hardness, TDS, Ca, Mg, SO_4

- Fluoride
- Nitrate
- Hardness, TDS
- Ca, Mg, Cl

Adverse Effects of Impurities

- Eutrofication of the waterbody
- Taste, discolouration and corrosion of pipes fittings and utensils
- Promotes iron bacteria
- Corrosion in water supply system
- Carcinogenic effect
- Toxic effect
- Formation of chlorophenols with chlorine
- Imparts unpleasant taste and odour after chlorination
- Water-borne diseases

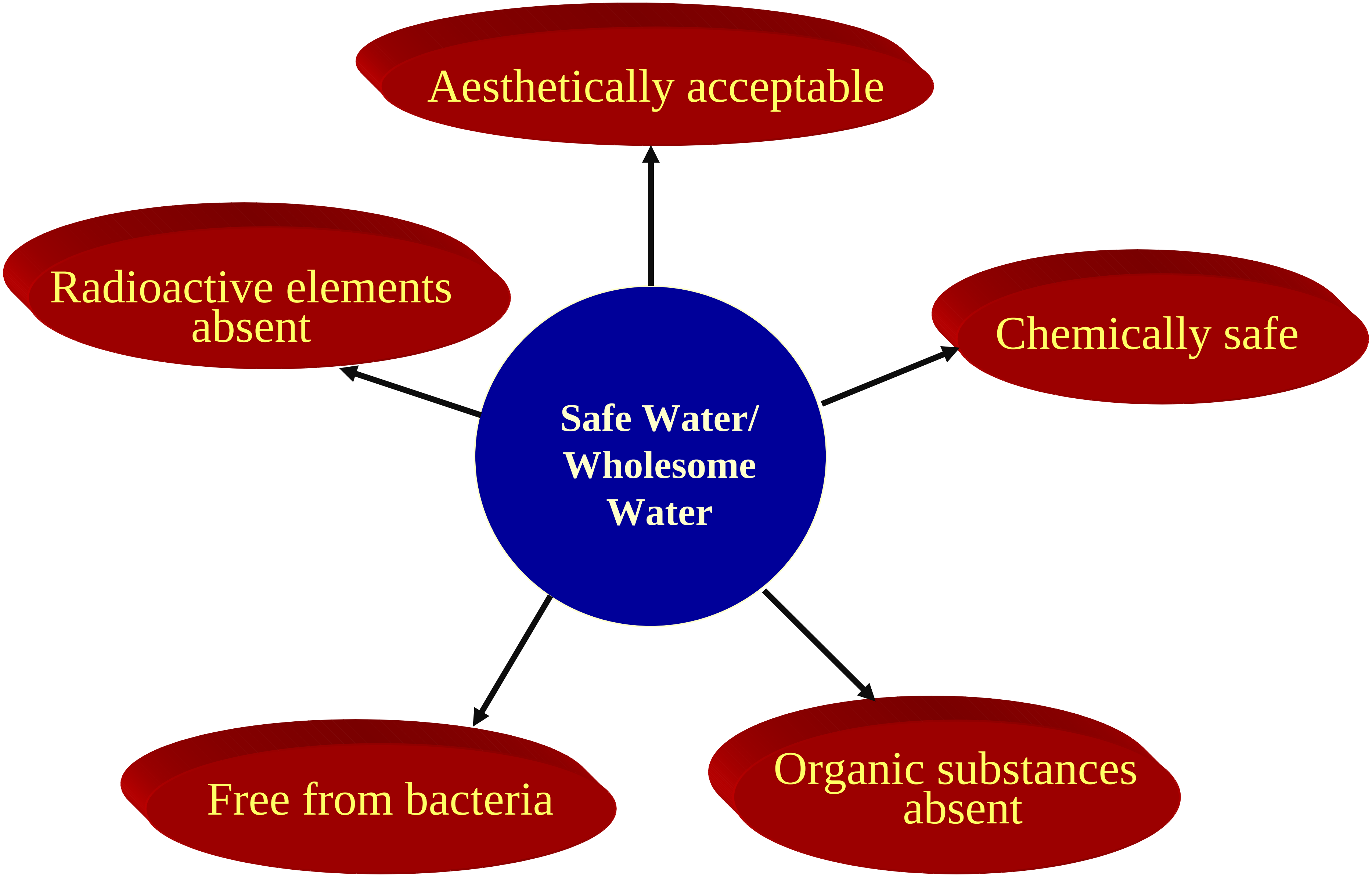
- Zoo & Phyto, Phosphate, Nitrate
- Iron, Mn, Cu, Zn, Alkalinity
- Fe & Mn
- pH, Cl
- Cr, As
- Cd, Pb, Hg
- Phenols
- Oil & grease
- Bacteria & viruses

Contamination / Pollution

- ✓ During its traverse water picks up impurities in varying amounts
- ✓ Gases from atmosphere
- ✓ Inorganic and organic salts from top soil and geological strata
- ✓ During its traverse water get contaminated by inorganic and organic salts sometimes beyond desirable limits

Pollution

- ✓ Presence of undesirable substances in the quantities which are harmful to man vegetation or property is referred to as pollution
- ✓ Quality of water depends upon quality and quantity of inorganic and organic salts present in water



Safe Water/ Wholesome Water

- Aesthetically acceptable
- Chemically safe
- Organic substances absent
- Free from bacteria
- Radioactive elements absent

Water Quality Assessment

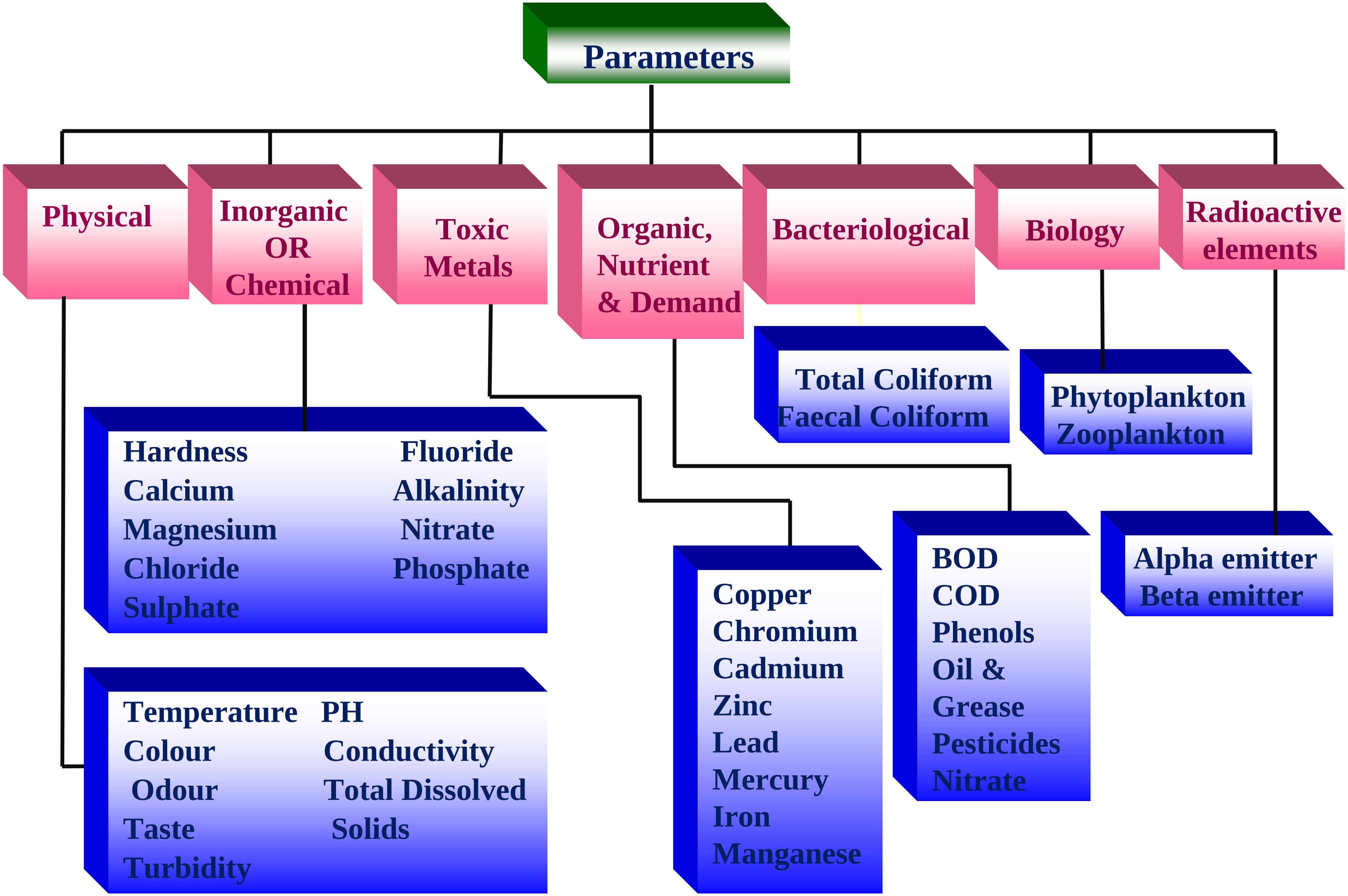
- To measure concentration of the constituents in quantity for characterisation of water for different uses
- Of the various parameters in potable water few are objectionable even when present in very small quantity
- Others if only present in unusual quantities as to relegate the water from the potable to the unusable class
- The analyst familiar with water quality characterisation will often select parameters to be measured based on experience and intuition

Water Analysis - Key Factors

Factors

- Sampling
- Proper Labeling
- Selection of Parameters
- Precision and accuracy of method selected
- Reporting the values
- Preservation

Water Quality Assessment : Potable & Industrial Uses



Parameters

Physical

Inorganic OR Chemical

Toxic Metals

Organic, Nutrient & Demand

Bacteriological

Biology

Radioactive elements

Total Coliform
Faecal Coliform

Phytoplankton
Zooplankton

Hardness
Calcium
Magnesium
Chloride
Sulphate

Fluoride
Alkalinity
Nitrate
Phosphate

BOD
COD
Phenols
Oil & Grease
Pesticides
Nitrate

Alpha emitter
Beta emitter

Copper
Chromium
Cadmium
Zinc
Lead
Mercury
Iron
Manganese

Temperature
Colour
Odour
Taste
Turbidity

PH
Conductivity
Total Dissolved Solids

Water Quality Consideration

- Irrigation
  - ✓ pH
  - ✓ Conductivity
  - ✓ Sodium & Potassium
  - ✓ Nutrients
  - ✓ Specific compounds

- Industries
  - ✓ As per specific requirement

- Domestic Consumption
  - ✓ As per BIS Standards

- Water Bodies
  - ✓ As per CPCB guidelines

Classification of Inland Surface Waters (CPCB Standards)

S. No.	Characteristics	A’	B’	C’	D’	E’
1.	Dissolved Oxygen, mg/L, Min	6	5	4	4	-
2.	Biochemical Oxygen Demand, mg/L, Max	2	3	3	-	-
3.	Total Coliform Organisms*, MPN/100 mL, Max	50	500	5000	-	-
4.	Total Dissolved Solids, mg/L, Max	500	-	1500	-	2100
5.	Chlorides (as Cl), mg/L, Max	250	-	600	-	600
6.	Colour, Hazen Units, Max	10	300	300	-	-
7.	Sodium Absorption Ratio, Max	-	-	-	-	26
8.	Boron (as B), mg/L, Max	-	-	-	-	2
9.	Sulphates (as SO_4), mg/L, Max	400	-	400	-	1000
10.	Nitrates (as NO_3), mg/L, Max	20	-	50	-	-
11.	Free Ammonia (as N), mg/L, Max	-	-	-	1.2	-
12.	Conductivity at 25^{o}C, micromhos/cm, Max	-	-	-	1000	2250
13.	pH value	6.5-8.5	6.5-8.5	6.5-8.5	6.5-8.5	6.5-8.5
14.	Arsenic (as As), mg/L, Max	0.05	0.2	0.2	-	-
15.	Iron (as Fe) , mg/L, Max	0.3	-	50	-	-
16.	Fluorides (as F), mg/L, Max	1.5	1.5	1.5	-	-
17.	Lead (as Pb), mg/L, Max	0.1	-	0.1	-	-
18.	Copper (as Cu), mg/L, Max	1.5	-	1.5	-	-
19.	Zinc (as Zn), mg/L, Max	15	-	15	-	-

- If the coliform count is found to be more than the prescribed tolerance limits, the criteria for coliforms shall be satisfied if not more than 20 percent of samples show more than the tolerance limits specified, and not more than 5 percent of samples show values more than 4 times the tolerance limits. Further, the faecal coliform should not be more than 20 percent of the coliform. Source : Indian Standard (IS:2296-1982).

A’	Drinking water source without conventional treatment but after disinfection
B’	Outdoor bathing (organised)
C’	Drinking water source with conventional treatment followed by disinfection
D’	Propagation of wild life, fisheries
E’	Irrigation, industrial, cooling, controlled waste disposal

Dissolved Oxygen

- All living organisms depend upon oxygen to maintain the metabolic processes that produce energy for growth and reproduction
- Dissolved oxygen is important in precipitation and dissolution of inorganic substances in water

Need

- To assess quality of raw water
- To check on pollution
- Determination of biological changes by aerobic or anaerobic organisms
- D.O. is the basis of BOD test to evaluate pollution potential of wastes
- All aerobic biological wastewater treatment processes
- Important factor in corrosion.

Methodology

The Winkler method with Azide modification

Principle

- Oxygen present in sample oxidizes the divalent manganous to its higher valency which precipitates as a brown hydrates oxide after addition of NaOH and KI
- Upon acidification, manganese revert to divalent state and liberates Iodine from KI equivalent to D.O. content in the sample
- The liberated Iodine is titrated against standard (N/40) solution of Sodium thiosulphate using starch as an indicator.

Procedure

- Collect sample in BOD bottle
- 2 ml $MnSO_4$+ 2 ml Alkali iodide-azide+close stopper
- Mix well + allow the ppt to settle
- Add 2 ml concentrated H_2SO_4 + mix well till ppt dissolves
- Take 203 ml (Correspond to 200 ml) sample in a conical flask+titrate against Sodium thiosulphate (0.025 N) till pale yellow colour + starch + titrate till blue to colourless

Calculation

- 1 ml of 0.025N $Na_2S_2O_3$ = 0.2 mg of O_2
- D.O. in mg/l = $\frac{(0.2 \times 1000) \times \text{ml of thiosulphate}}{200}$

Results : D.O. mg/l

Interferences

- Ferrous ion
- Ferric ion
- Nitrite
- Microbial mass
- High suspended solids

To reduce interferences, modification in the estimation procedures are suggested

- Alsterberge azide : Nitrite, higher concentration of ferric ions
- Redeal Stewart : Ferrous ion
- Alum / Flocculation : High suspended solids
- Copper Sulphate Sulfamic acid flocculation : Biological flocs
- Alkaline Hypochlorite : Complex of sulphur compound

Biochemical Oxygen Demand

A bioassay test, involving measurement of oxygen consumed by micro-organisms while stabilizing biologically decomposable organic matter under aerobic conditions

Need

- ➢To determine the pollution load of waste water
- ➢The degree of pollution in water sources
- ➢Self purification capacity of sources
- ➢Designing of treatment facilities
- ➢Efficiency of waste water treatment methods

Methodology

Principle

- The BOD test is based upon determinations of dissolved oxygen
- It can be measured directly
- In general, a dilution procedure is applied.

Procedure

Preparation of dilution water

- Aerate the required volume of D.W. by bubbling compressed air for 1-2 days to attain D.O. saturation
- Add 1 ml each per litre of dilution water
  Phosphate buffer
  Magnesium sulphate
  Calcium chloride
  Ferric chloride
- Mix well
- In case, waste not expected sufficient bacterial population, add seed (2 ml settle sewage / litre of dilution water)

Determination of D.O.

i) Samples and ii) Blank, on initial and after 5 days

- 2 ml $MnSO_4$ + 2 ml Alkali-iodide-azide+stopper immediately
- Mix well + allow the ppt. to settle
- Add 2 ml concentrated H_2SO_4 + mix well till ppt. dissolve
- Take 203 ml (correspond to 200 ml) sample in a conical flask
- Titrate against sodium thiosulphate (0.025 N) till pale yellow colour + starch solution + blue colour + titrate till colourless

Observations

D_0 = D.O. in sample on 0th day

D_1= D.O. in sample on 5th day

C_0 = D.O. in Blank on 0th day

C_1 = D.O. in Blank on 5th day

$C_0 – C_1$ = D.O. depletion in dilution water alone

$D_0 – D_1$ = D.O. depletion in sample + dilution water

$(D_0 - D_1) - (C_0 – C_1)$ = D.O. depletion due to microbes

Calculation

1 ml of 0.025 N sodium thiosulphate = 0.2 mg of Oxygen

$$\text{D.O. in mg/l} = \frac{(0.2 \times 1000) \times \text{ml of thiosulphate}}{200}$$

B.O.D. in mg/l $(D_0\text{-}D_1) - (C_0\text{-}C_1)$ mg **X** Decimal fraction of sample used

Results

B.O.D. 5 days at 20^0 C = mg/l

Interferences

- Ferrous ion
- Ferric ion
- Nitrate
- Microbial mass
- High suspended solids
  - Lack of nutrients in dilution water
  - Lack of acclimated seed organisms
  - Presence of heavy metals
  - Presence of toxic materials

Bacteriological Analysis

Bacteria

Single cell microscopic organisms lacking chlorophyll

Coliform group

Contamination

- **Insanitary condition of surrounding area**
- **Unhygienic practices**
- **Discharge/seepage of sewage and domestic wastewater**

Need

- **Impact on water quality**
- **Potability for human consumption**
- **To prevent water-borne diseases**
- **To assess the quality of raw and treated water**
- **Specially to detect Faecal Contamination**

Bacteriological analysis : mainly includes estimation of

- **Total coliforms**
- **Faecal coliforms**

Methodology

Approved techniques generally used as per "Standard Methods for the examination of water and wastewater"

- **.Membrane Filter (MF)**
- **Multiple Tube Dilution (MTD)**

MF-technique

Principle

Biochemical reactions are used to detect the various groups of micro-organisms

MF-technique - Merits

- **Results in 24 hours (MTD - 48 to 96 hours)**
- **Larger volume of samples can be tested (MTD - less volume)**
- **Results with greater precision (MTD - MPN)**
- **Require less laboratory space (MTD – More space)**
- **Easy processing (MTD – Tedious)**
- **Useful during normal and emergencies (MTD – Difficult in emergencies)**

Limitations : Samples with more turbidity

Colour

- Coloured water is not acceptable for drinking (Aesthetic as well as toxicity reasons)
- Industrial wastewater require colour removal before discharge into water courses

Definition

- The term colour means true colour that is the colour of water from which turbidity has been removed. True colour of water is due to dissolved material
- Apparent colour is due to suspended matter as well as due to substances on solution removed by filtration

Unit for Measurement of colour

- Unit for colour measurement is based on platinum cobalt scale

Contd...

Methods for Colour Measurement

Visual Comparison Method

- Colour of the sample is determined by visual comparison with known concentration of coloured colutions prepared by diluting stock platinum cobalt solution
- OR properly calibrated glass coloured disk is used for comparison
- This method is useful for potable water and water in which colour is due to naturally occuring materials
- This method is not applicable to most highly coloured industrial wastewater

Spectrophotometric Method

- This method is applicable to potable and waste both domestic and industrial
- In this method light absorbed or transmitted is measured at dominant wavelength of a particular hue of sample
- Spectrophotometer should have an effective operating range from 400 to 700 nm before measurement remove turbidity either by filtration or by centrifuging
- Colour hues for dominant wavelengths ranges are

Contd...

Conductivity Meter

Definition

Measure of the ability of aqueous solution to carry an electric current

- Concentration of ions
- Mobility
- Valency
- Temperature

Conductivity Measurement

Conductance(G)

∝ Area (A)
∝ 1/Distance (L)

$G = K(A/L)$
$A = 1cm^2$
$L = 1$ cm
$G = K$

K is called conductivity or specific conductance

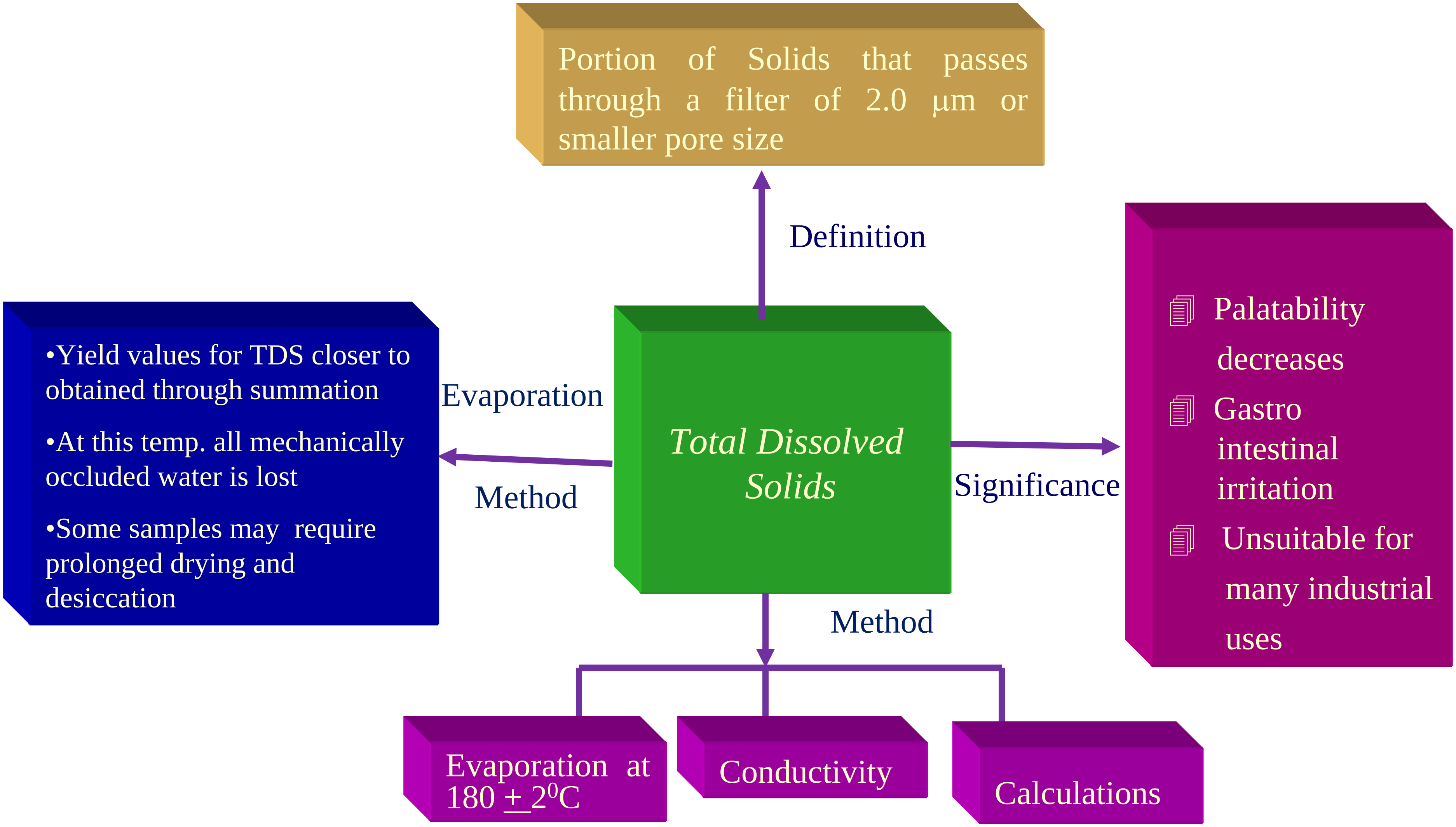
Conductance is measured between two spacially fixed and chemically inert platinum electrodes

Unit

μ mho/cm
or
μs/cm

Significance

- Degree of Minerilization
- Effect on plants and animals
- Check purity of distilled water
- Monitoring seasonal variation of raw water or wastewater
- Determine sample size for chemical analysis
- Check results of chemical analysis
- Estimate TDS



Portion of Solids that passes through a filter of 2.0 µm or smaller pore size

Definition

Total Dissolved Solids

Evaporation

Method

•Yield values for TDS closer to obtained through summation

•At this temp. all mechanically occluded water is lost

•Some samples may require prolonged drying and desiccation

Significance

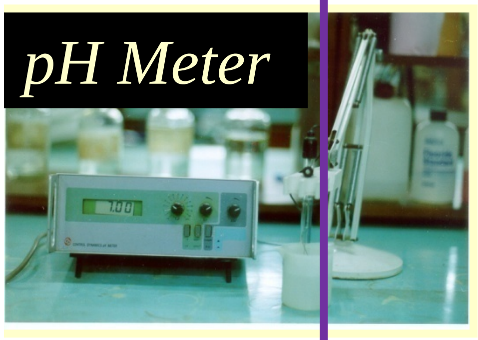
- Palatability decreases
- Gastro intestinal irritation
- Unsuitable for many industrial uses

Method

Evaporation at 180 $\underline{+}$ 2^0C

Conductivity

Calculations



pH Meter

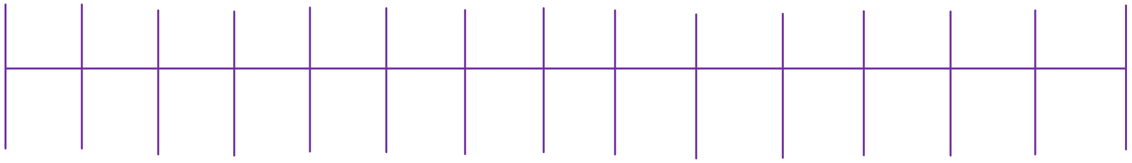
pH
Hydrogen Ion
Exponent
6.5 - 8.5

Definition

- **pH** = - $\log_{10} [H^+] = \log_{10} 1/[H^+]$ OR $[H^+] = 10^{-pH}$

  This method has advantage because all states of acidity and alkalinity of solutions with respect to hydrogen and hyroxide ions can be expressed by a series of positive numbers between 0 to 14

$[H^+]$	(10^{0})	10^{-1}	10^{-2}	10^{-3}	10^{-4}	10^{-5}	10^{-6}	10^{-7}	10^{-8}	10^{-9}	10^{-10}	10^{-11}	10^{-12}	10^{-13}	10^{-14}
pH	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14



pOH	14	13	12	11	10	9	8	7	6	5	4	3	2	1	0
$[OH^-]$	10^{-14}	10^{-13}	10^{-12}	10^{-11}	10^{-10}	10^{-9}	10^{-8}	10^{-7}	10^{-6}	10^{-5}	10^{-4}	10^{-3}	10^{-2}	10^{-1}	10^{-0}

Significance

- Chemical reactions depend on pH
- Water Supply and Waste Water Treatment
- Water Softening ,Precipitation., Coagulation, Disinfection, Corrosion Control,Alkalinity and CO_2 Measurement and fluoride activity

Measurement

- Electrometric method - Using pH meter and electrodes
- e.m.f. produced in glass electrode system varies linearly with pH
- pH meter is calibrated potentiometrically with electrode system using standard buffers having assigned values so that pH = - $\log_{10} [H^+]$

Nitrate

Sources

- Fertilizers and manure
- Decayed vegetable
- Animal feedlots
- Municipal wastewater and sludge disposal to land
- Industrial discharges
- Leachates from refuse dumps
- Septic systems and
- N-fixation from atmosphere by bacteria and lightning

Chloride

Presence in Natural Waters

- Dissolution of salt deposits
- Discharges of effluents
- Oil well operations
- Sewage discharges
- Irrigation drainage
- Sea water intrusion in coastal areas

Chloride

Methodology : An Argentometric Method

- Principle

Chloride is determined in a natural or slightly alkaline solution by titration with standard silver nitrate, using potassium chromate as an indicator. Silver chloride is quantitatively precipitated before red silver chromate is formed.

$$\text{Chloride mg/L} = \frac{(A-B) \times N \times 35.45 \times 1000}{\text{ml sample}}$$

Where A = ml $AgNO_3$ required for sample

B = ml $AgNO_3$ required for blank

N = Normality of $AgNO_3$ used

Methods for Nitrate Estimation

A) Ultraviolet Spectrophotometric Method

- Useful for uncontaminated natural waters and potable water supplies that have low organic content
- Follows Beer's law upto 11 mg/L as N
- Interferences
  - Dissolved Organic Matter
  - Surfactants
  - Nitrite and Hexavalent Chromium
- Procedure
  - Filter the sample
  - Add 1 ml of 1N HCl per 50 ml of sample
  - Read absorbance or tranmittance at 220 nm and 275 nm
  - Set 0 absorbance or 100% tranmittance with distilled water

B) Nitrate Electrode Method

- Useful for Nitrate concentration range of 0.14 to 1400 mg/L NO_3-N
- Interferences
  - Chloride and bicabonate with weight ratios to NO_3-N >10 or >5 respectively
  - $NO_{2,}$ CN, Sulphide, Br, I, Chlorite and Chlorate

C) Phenoldisulphonic Acid (PDA) Method

- **Nitrate reacts with Phenoldisulphonic acid to produce nitro derivatives that in alkaline solution rearranges its structure to form yellow colored compound with characteristics that follows Beer's law**
- **Chloride interferes seriously which can be overcome by precipitation of chloride with Ag^+ as AgCl**

Fluoride

Significance

Dual significance in water

High concentration of F^- causes dental Fluorosis

Concentration < 0.8 mg/L results in dental Carries

Essential to maintain F^- concentration between 0.8 mg/L to 1.0 mg/L in drinking water

Fluoride

Methods

Colorimetric SPADNS Method

Principle: Under acidic conditions fluorides (HF) react with zirconium SPADNS solution and the lake (colour of SPADNS reagent) gets bleached due to formation of ZrF_6 . Since bleaching is a function of fluoride ions, it is directly proportional to the concentration of fluoride. It obeys Beers law in a reverse manner.

Ion Selective Electrode Method

Principle: The fluoride sensitive electrode is of the solid state type, consisting of a lanthanum fluoride crystal; in use it forms a cell in combination with a reference electrode, normally the calomel electrode. The crystal contacts the sample solution at one face and an internal reference solution at the other. A potential is established by the presence of fluoride ions across the crystal which is measured by a device called ion meter or by any modern pH meter having an expanded millivolt scale.

Calculate mg F^- / L present in the sample using standard curve

Sulphate

Significance

- Occurs in natural water
- High concentration of Sulphate laxative effect (enhances when sulphate consumed with magnesium)
- Problem of scaling in industrial water supplies
- Problem of odour and corrosion in wastewater treatment due to its reduction to H_2S

Sulphate

Method

Spectorphotometric Method

Principle: Sulfate ions are precipitated as $BaSO_4$ in acidic media (HCl) with Barium Chloride. The absorption of light by this precipated suspension is measured by spectrophotometer at 420 nm or scattering of light by Nephelometer

Calculate

$$\text{mg / L } SO_4 = \frac{\text{mg } SO_4 \times 1000}{\text{ml sample}}$$

Ammonia

- Ammonia is present naturally in surface and wastewaters. Its concentration is generally low in ground waters because it adsorbs in soil particles and clays and is not leached readily from soils.
- It is produced largely by de-amination of organic nitrogen containing compounds and by hydrolysis of urea
- In the chlorination of water, chlorine reacts with ammonia to form mono and dichloramines (combined residual chlorine)
- Ammonia concentration in water vary from less than 10μg in some natural surface and ground waters to more than 30 mg/L in some wastewaters

Methods for Ammonia Estimation

A) Nesslerization Method

- Direct nesslerization method is useful for purified natural water and highly purified wastewater effluents with very light color and having NH_3-N concentrations more than 20 μg/L
- Applicable to domestic wastewater only when errors of 1 to 2 mg/L are acceptable
- The graduated yellow to brown colors produced by nessler-ammonia reaction absorb strongly over wide wavelength range
- Low ammonia concentration of 0.4 to 5 mg/L can be measured with acceptable sensitivity in wavelength region from 400 to 425 nm with 1cms light path
- A light path of 5 cm extends measurements of ammonia concentrations range of 5 to 60 μg/L

- **The reddish brown color at ammonia levels approaching 10 mg/L may be measured in the wavelength region of 450 to 500 nm**
- **Thus, the proper selection of light path and wavelength permits the photometric determination of ammonia concentrations over a considerable range**
- **Carefully prepared nessler reagent may respond under optimum conditions to as low as 1μg NH_3-N/ 50mL (100μg/L). However, reproducibility below 100 μg/L may be erratic**
- **Interferences**
- **Turbidity, color , magnesium and calcium**

B) Ammonia Selective Electrode Method

- **The ammonia selective electrode uses a hydro-phobic gas permmeable membrane to separate the sample solution from an electrode internal solution of ammonium chloride**
- **Dissolved ammonia is converted to $NH_{3(aq)}$ by raising pH to above 11 with a strong base, which diffuses through membrane and changes the internal solution pH that is sensed by a pH electrode**
- **The fixed level of chloride in the internal solution is sensed by a chloride ion-selective electrode that serves as the reference electrode**
- **Applicable to the measurement of 0.03 to 1400 mg NH_3-N/L in potable and surface waters and domestic and industrial wastes**
- **High concentrations of dissolved ions affect the measurements but color and turbidity do not.**
- **Interference**

- **Amines are a positive interference**
- **Hg and silver interfere by complexing with ammonia**

C) Titrimetric Method

- The method is used only on samples that have been carried through preliminary distillation
- Titrate ammonia in distillate using standard 0.02N Sulphuric acid with boric acid indicator solution

Phosphates

- Phosphate occurs in traces in many natural waters, and often in appreciable amounts during periods of low biologic productivity. Waters receiving raw or treated sewage agricultural drainage, and certain industrial waters normally contain significant concentrations of phosphate.

Methods for Phosphorus Estimation

A) Vanadomolybdophosphoric Acid Method

- In a dilute orthophosphate solution, ammonium molybdate reacts under acid conditions to form a heteropoly acid. In the presence of vanadium, yellow vanadomolybdophosphoric acid is formed. The intensity of yellow color is proportional to phosphate concentration
- Minimum detectable concentration is 0.2 mg P/L in 1 cm cell
- Interferences
- Silica and arsenate cause positive interference if the sample is heated
- Negative interferences are caused by fluoride, thorium, bismuth, sulphide, thiosulfate, thiocyanate or excess molybdate
- Blue color is caused by ferrous iron but this does not affect results if its concentration is less than 100 mg/L
- Sulphide interference may beremoved by oxidation with bromine water

Procedure

- Sample pH adjustment if pH > 10
- Removal of excessive color by shaking with activated carbon
- Color development with vanadate-molybdate reagent
- Measurement of color absorbance at wavelength of 400 to 490 nm

B) Stannous Chloride Method

- **Molybdophophoric acid is formed and reduced by stannous chloride to intensely colored molybdenum blue**
- **This method is more sensitive than above method and minimum detectable concentration is about 3 μg P/L**
- **Interferences**
- **Silica and arsenate cause positive interference if the sample is heated**
- **Negative interferences are caused by fluoride, thorium, bismuth, sulphide, thiosulfate, thiocyanate or excess molybdate**
- **Blue color is caused by ferrous iron but this does not affect results if its concentration is less than 100 mg/L**
- **Sulphide interference may beremoved by oxidation with bromine water**
- **Procedure**
- **Sample pH adjustment if pH > 10**
- **Color development with molybdate reagent**
- **Measurement of color absorbance at wavelength of 690 nm**

Limit of Iron and Manganese in Drinking Water

- As per WHO guidelines for domestic water, iron should not exceed the limit of 0.3 mg/l
- Above 200mg/l iron is toxic to human health
- Manganese concentration as per WHO guideline is 0.05 mg/l
- However average manganese level in drinking water range from 5 to 25 ug/l
- At concentration exceeding 0.15 mg/l, manganese imparts undesirable taste

Iron and Manganese

- Presence of excess of iron and manganese in water causes discoloration, turbidity and deposits
- Iron and manganese bearing water have astringent metallic or bitter taste
- Precipitation of iron and manganese imparts colour to water from yellow to brownish black, which becomes objectionable to consumers
- Manganese concentration ranging from 8-14 mg/l is toxic to human
- Excess of iron facilitates growth of iron bacteria which causes blocking of pipes, meters etc.

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Methods for Detection of Iron and Manganese in Water

- Atomic Absorption spectrophotometer (AAS)

- Inductively Coupled Plasma (ICP)

- Colorimetric method

  In colorimetric method iron is detected at wavelength 510 nm and manganese is detected at 525 nm.

1. Iron:- Phenanthroline method
2. Manganese:- Persulphate method
   Periodate method

Determination of Metals

Inductively Coupled Plasma-Atomic Emission Spectrometer

Optima 4100 DV ICP-OES

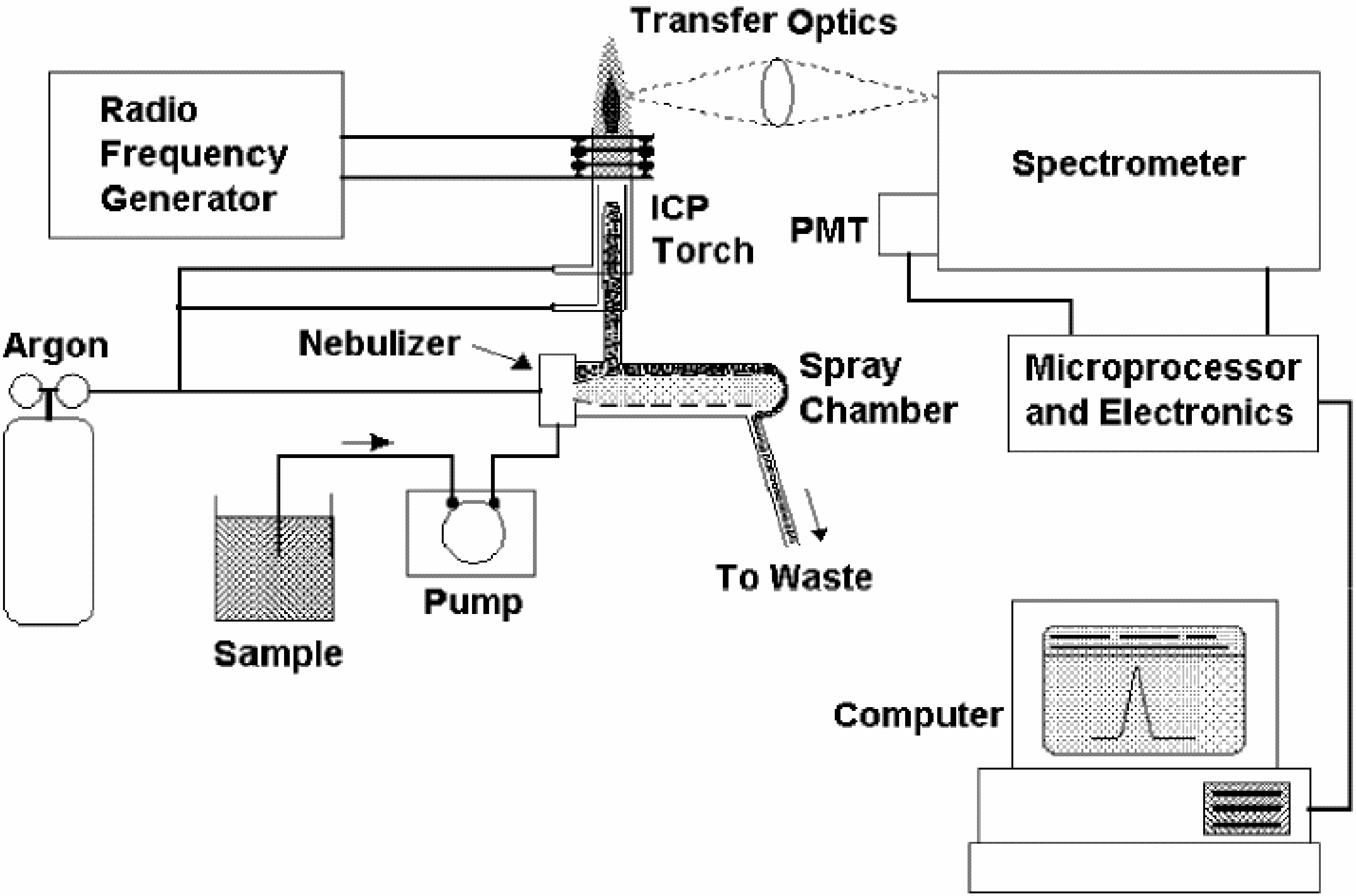
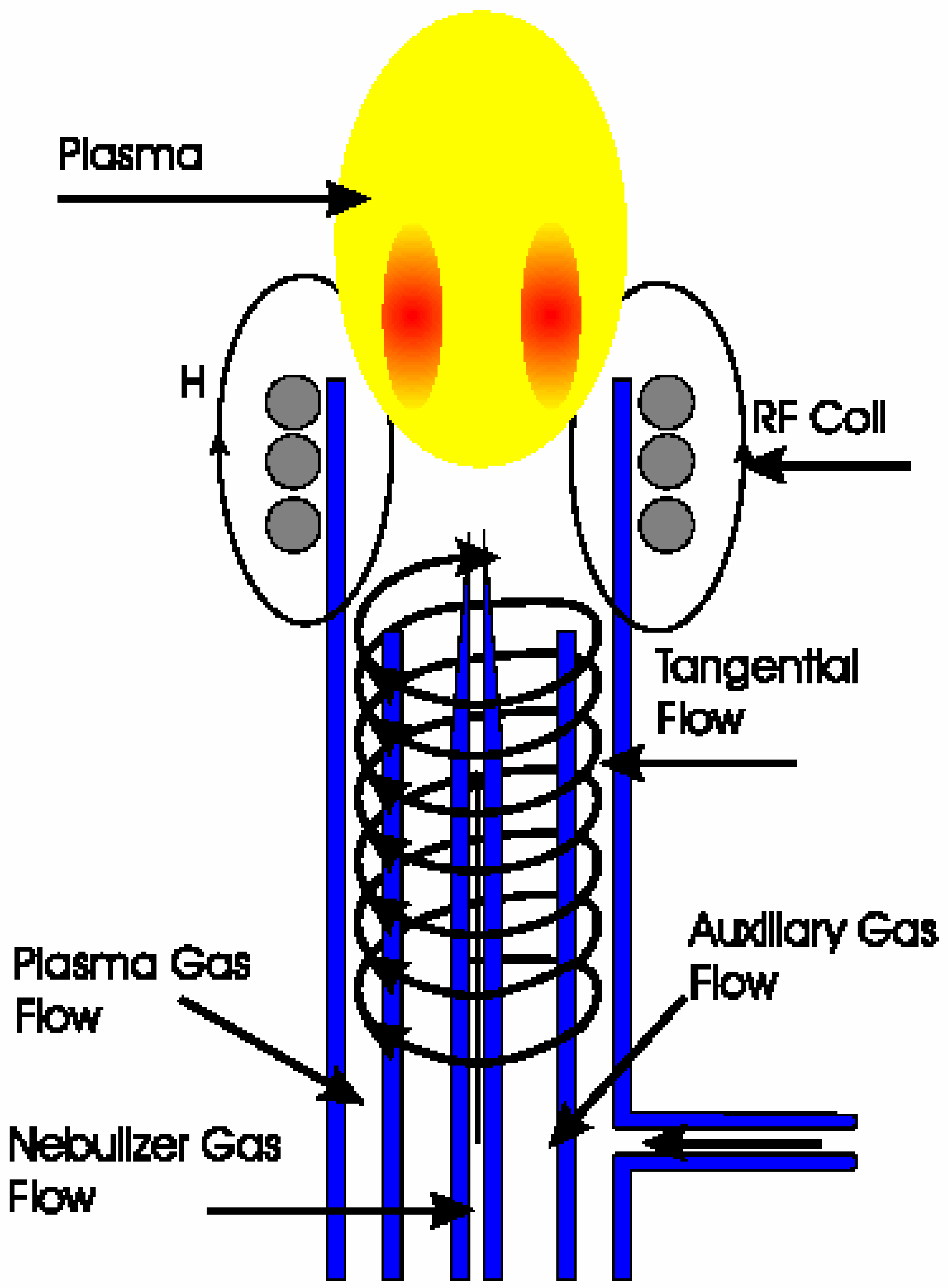


Figure 3-1. Major components and layout of a typical ICP-OES instrument.



ICP TORCH

- Radio frequency

27-49 MHz

700-1599 W

- Gas- Argon

1. Sample Preparation: Some samples require special preparation steps incuding treatment wit acids, heating, and microwave digestion.

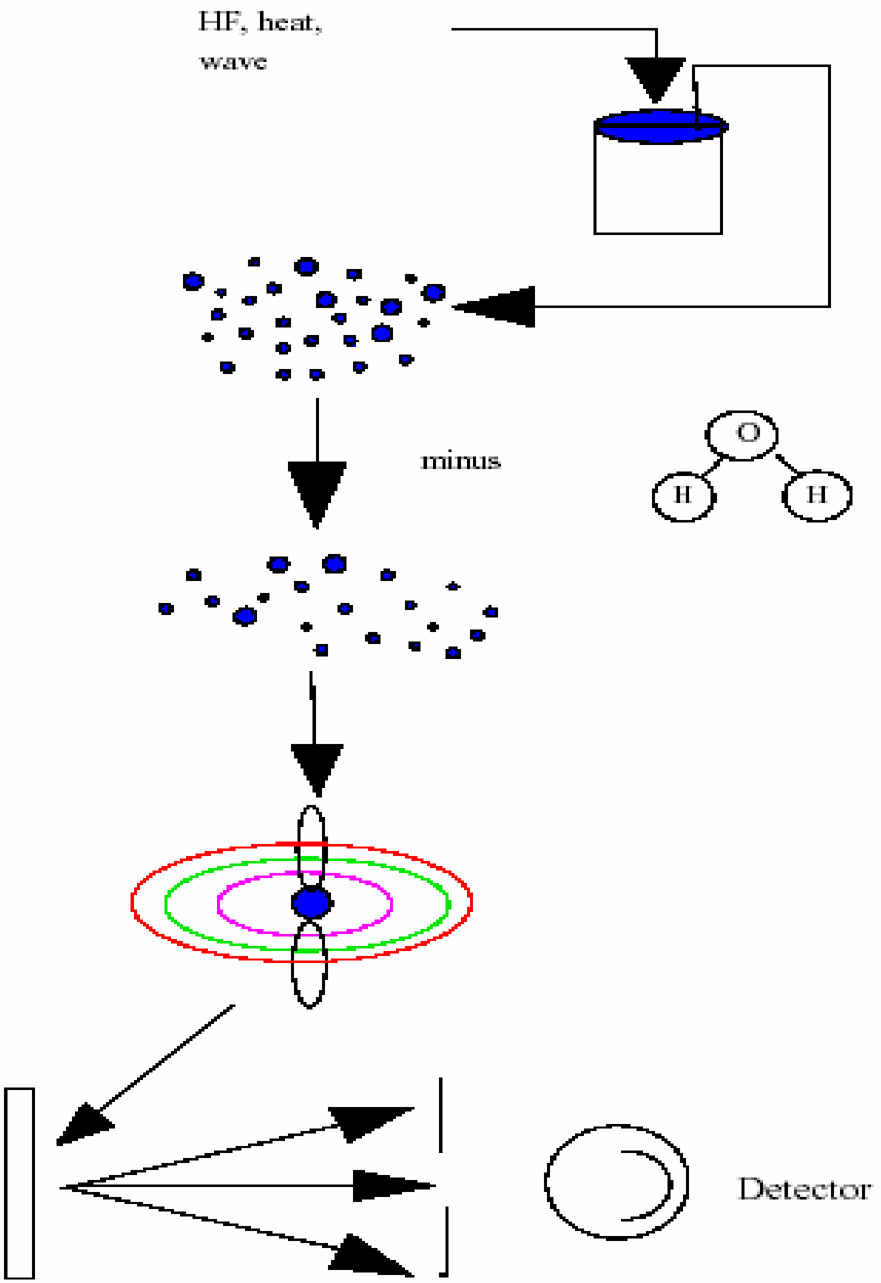
2. Nebulization: Liquid converted to aerosol.

3. Desolvation/Volatization:
Water is driven off, and remaining solid and liquid portions are converted to gases.

4. Atomization: Gas phase bonds are broken, and only atoms are present. Plasma temperature and inert chemical environment are important at this stage.

5. Excitation/Emission: Atoms gain energy from collisions and emit light of a characteristic Wavelength.

6. Separation/Detection: A grating dispersers light that is quantitatively measured.



Steps involved in the analysis of aqueous samples by ICP-AES

Procedure

Instrument set up

- Warm up for 30 min
- Check the alignment of plasma torch
- Make Cu/Mn ratio adjustment
- Calibrate instrument using calibration standards and blank
- Aspirate the standard and blank for 15s
- Rinse with calibration blank for at least 60s to eliminate any carryover from previous standards
- Ensure the concentration values within the 5% error

Analysis of samples

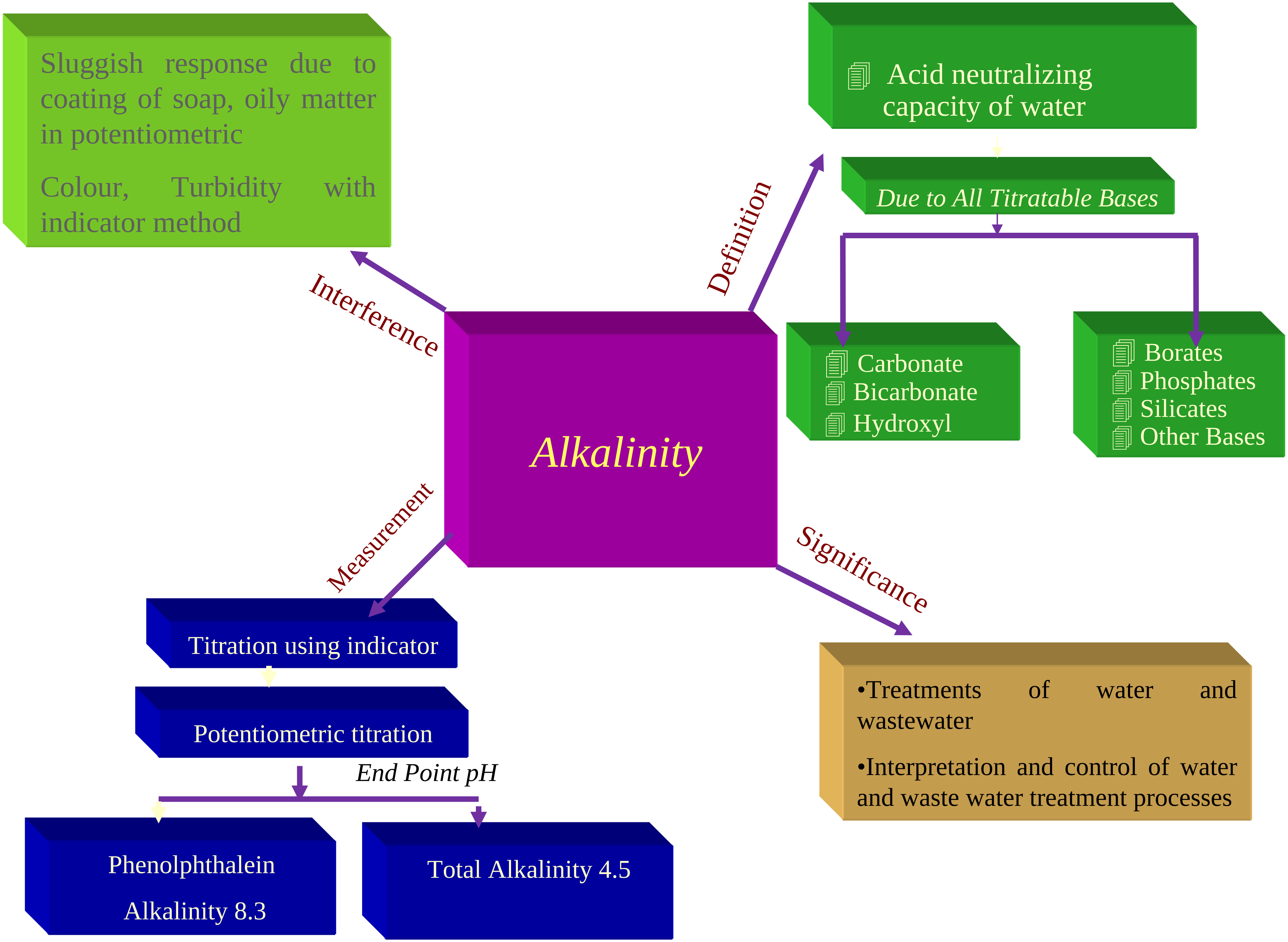
- Analysis the samples using calibration blank
- Analyse samples alternately with analyses of calibration blank
- Rinse at least for 60s
- Examine each analysis of the calibration blank to verify that carry over memory effect is no more
- Make appropriate dilutions of the sample to determine concentrations beyond the linear calibration

Instrument quality control

- Reanalyse one or more samples analysed just before termination of the analytical run
- Use this analysis to verify accuracy and validity of the calibration standards

Children with safe water

Thank You



Alkalinity

Interference

Sluggish response due to coating of soap, oily matter in potentiometric

Colour, Turbidity with indicator method

Definition

- Acid neutralizing capacity of water

Due to All Titratable Bases

- Carbonate
- Bicarbonate
- Hydroxyl

- Borates
- Phosphates
- Silicates
- Other Bases

Measurement

Titration using indicator

Potentiometric titration

End Point pH

Phenolphthalein Alkalinity 8.3

Total Alkalinity 4.5

Significance

- Treatments of water and wastewater
- Interpretation and control of water and waste water treatment processes

Turbidity

General

- Turbidity in water is caused by suspended matter such as

  Clay

  Silt

  Finely divided organic and inorganic matter

  Soluble coloured organic compounds

  Plankton and other microscopic organisms

Definition

- Turbidity is an expression of the optical property that causes light to be scattered and absorbed
- It is not possible to correlate turbidity with the weight concentration of suspended matter because light scattering properties of the suspended particulate matter depends upon size, shape and refractive index of the particulates

Contd...

Significance

- Clarity of water is important for human consumption
- Manufacturing processes such as beverages, and many food products need clear water

Unit for Turbidity Measurement

- Previously standard method for measurement of turbidity was based on the Jackson Cnadle turbidimeter
- The lowest turbidity value that can be measured by this instrument is 25 J.T.U.
- Need was felt to measure turbidity values even less than one unit because turbidities of treated water usually fall within the range of 0 to 1 unit
- Different types of secondary instruments were developed to measure turbidity values in low range

Contd...

- The results obtained with different types of secondary instruments frequently do not match closely with one another, even though the instruments are precalibrated against Jackson Candle turbidimeter, because of fundamental differences in optical systems
- Nephelometers developed for measuring low turbidities give comparatively good indications of the intensity of light scattered at right angles to the incident light, and are specified as the standard instrument for measurement of low turbidities
- The prescribed for turbidity measurement by Nephelometer is Nephelometric Turbidity Units (N.T.U.)
- Formazin polymer is used as the reference turbidity standard suspension
- Because there is no direct relationship between the intensity of light scattered at 90^{O} angle and Jackson candle turbidity there is no basis for the practice of calibrating a Nephelometer in terms of candle units
- However specific concentration of formazin polymer suspension defined as 40 NTU turbidity has an approximate turbidity of 40 JTU

Contd...

Method

Nephelometric

- Nephelometric method of turbidity measurement is based in a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension under the same conditions
- Higher the intensity of scattered light the higher the turbidity
- Turbidimeter with a tungston filament lamp as a light source for illuminating the sample and a photoelectric detector with a read out device is a system used for turbidity measurement by turbidimeter
- Meter is designed to prohibit stray light reaching to detector
- Short warm period is necessary to make the instrument free from significant drift
- Clear colourless glass tube is used for sample

Contd...

Hardness

Definition

- Total Hardness is defined as the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in mg/L
- Originally water hardness was under stood to be a measure of the capacity of eater to precipitated soap
- Soap is precipitated chiefly by calcium and magnesium ions present. Other prevalent cations also precipitate soap but they often are in complex forms and minimal concentration

Significance

- Scale formation in boilers, pipes and cooking utensils
- Adverse effect on domestic use
- Encrustation in water supply structure
- Cathartic and diuretic effect

Contd...

Type

- Carbonate hardness
- Non carbonate hardness

Methods of Analysis

Hardness by calculation

- Calcium can be estimated by AAS, ICP and EDTA titrimetric methods
- Magnesium can be estimated by AAS, ICP and Gravimatric method

Total Hardness by Calculation :

$$\text{mg } CaCO_3 / L = 2.497 \text{ [Ca mg/L]} + 4.118 \text{ [Mg mg/L]}$$

Contd...