

A PROJECT REPORT ON
“WATER ANALYSIS”



DISSERTATION SUBMITTED TO THE RANI CHANNAMMA
UNIVERSITY BELAGAVI, ON THE PROJECT WORK FOR THE
PARTIAL FULLFILLMENT OF THE DEGREE OF

BACHELOR OF SCIENCE
IN
CHEMISTRY

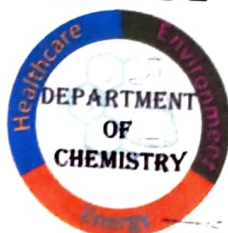
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CERTIFICATE

This is to certify that the dissertation entitled with the
“STUDY OF WATER ANALYSIS” for the award of the
degree of the Bachelor of the science in Chemistry is based on
research work carried out by **SWATI. MULLUR** under the
Department of Chemistry.

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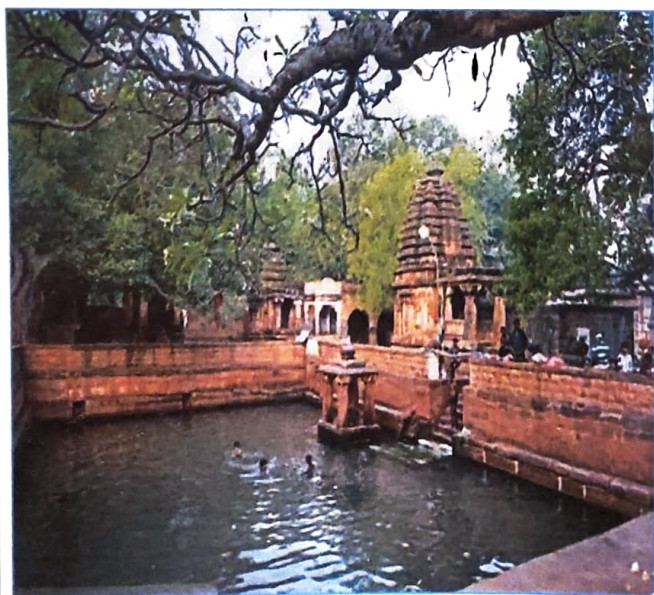
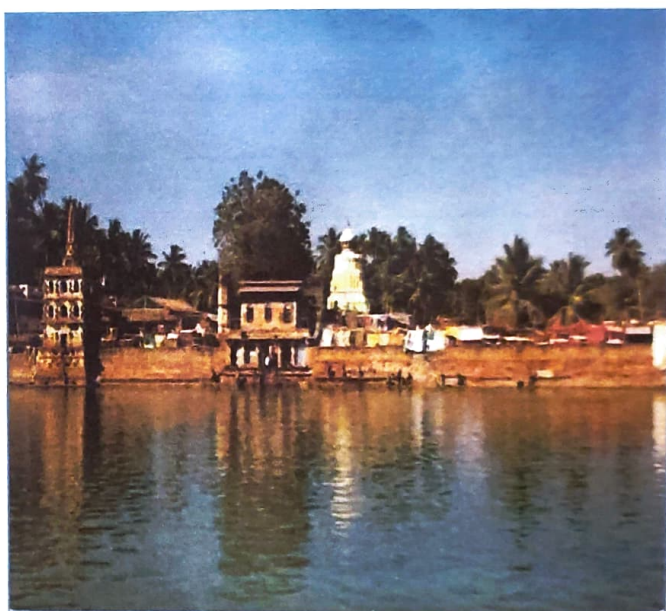
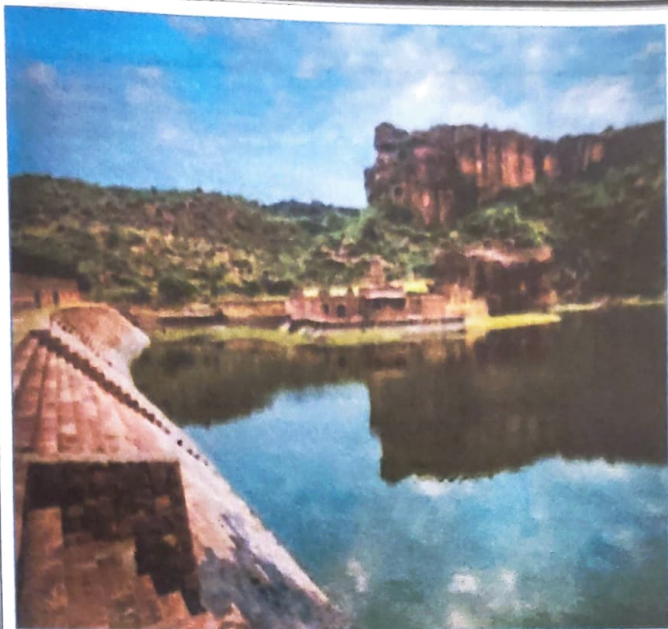


Contamination of pollution

- ✓ **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**

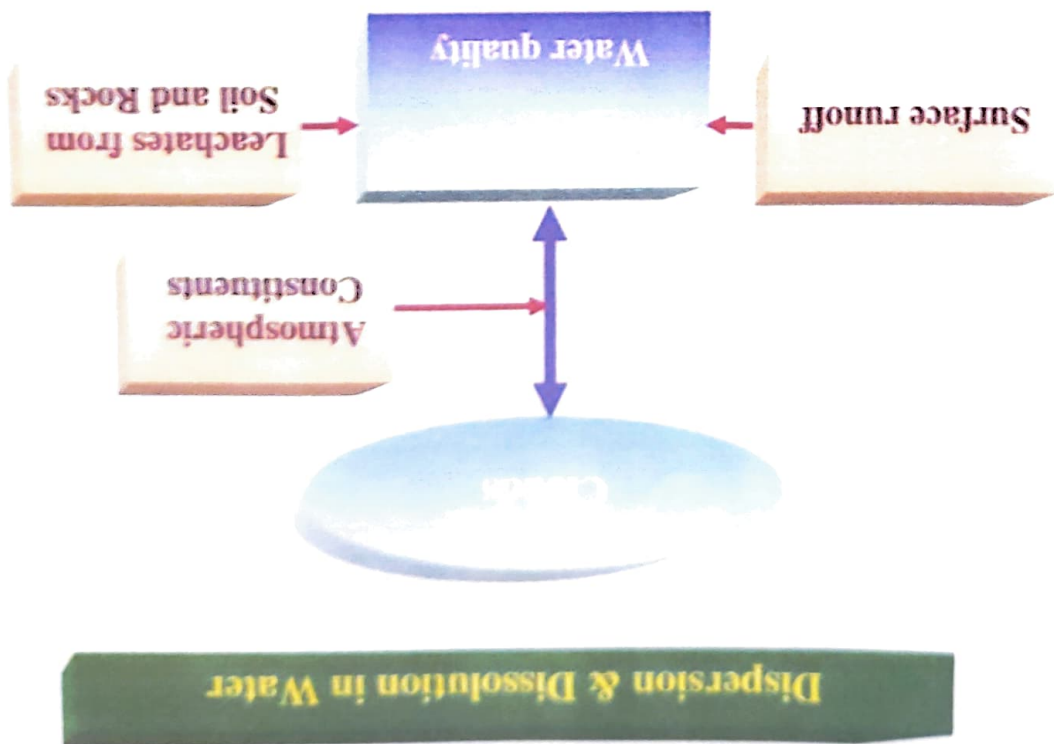
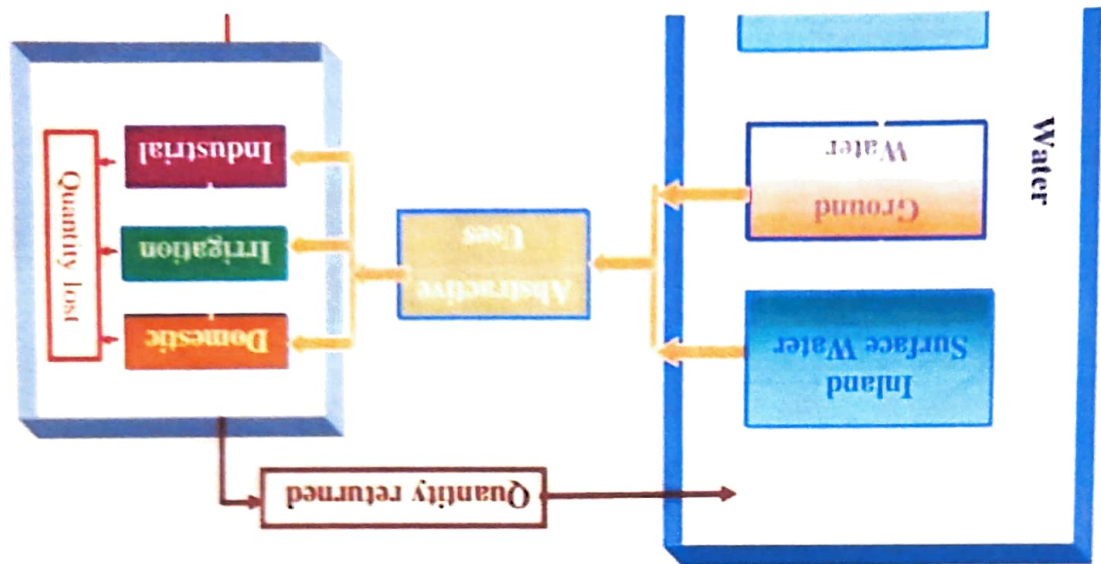


Water Quality Assessment

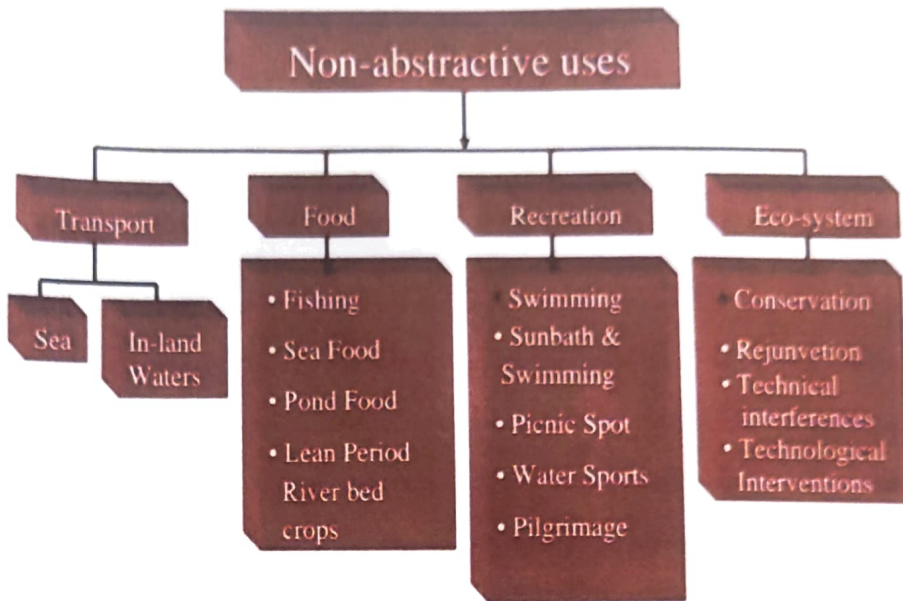
- ✓ To measure concentration of the constituents in quantity for characterisation of water for different uses
- ✓ Of the various parameters in potable water few are objectionable even when present in very small quantity
- ✓ Others if only present in unusual quantities as to relegate the water from the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

Water Quality Consideration

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



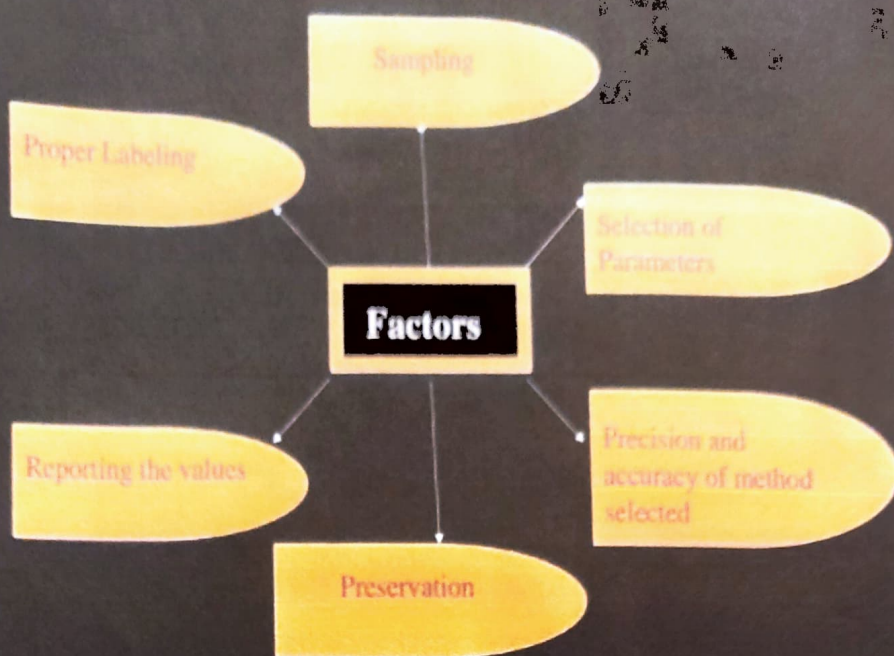
Water



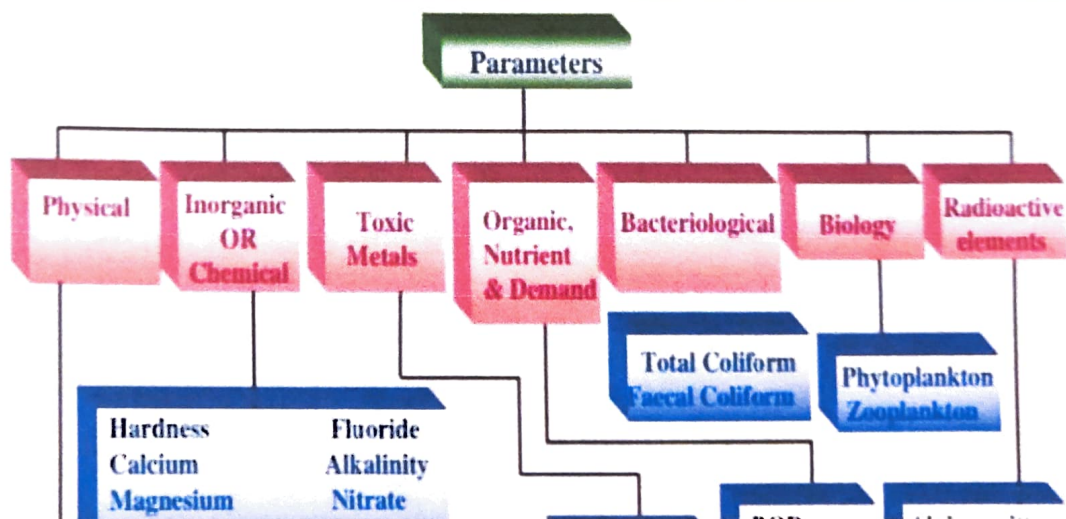
Adverse Effects of Impurities

Problems	Constituents Responsible
➤ Aesthetically not acceptable and Palatability decreases ➤ Health related problems ➤ affect mucous membrane ➤ gastro-intestinal irritation ➤ Dental and skeletal fluorosis ➤ Methaemoglobinemia	• Clay, Silt, Humus, Colour • pH • Hardness, TDS, Ca, Mg, SO₄ • Fluoride • Nitrate

Water Analysis - Key Factors



Water Quality Assessment : Potable & Industrial Uses



✓ Specific compounds

➤ **Industries**

✓ As per specific requirement

➤ **Domestic Consumption**

✓ As per BIS Standard

✓ 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 $\text{Na}_2\text{S}_2\text{O}_3$ = 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

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)

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

METHODS OF COLOUR MEASUREMENT

VISUAL COMPARISON METHOD

- Colour of the sample is determined by visual comparison with known concentration of coloured solutions 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 occurring materials
- This method is not applicable to most highly coloured industrial wastewater

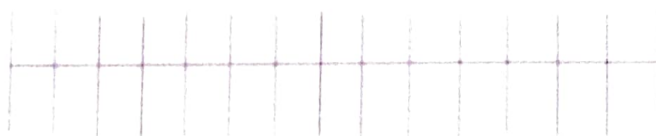


Definition

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

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

[H ⁺]	(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 CO₂ Measurement and fluoride activity

SPECTROPHOTOMETRIC METHOD

- 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

Nitrate

SOURCES:

- **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**

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.
- Chloride mg/L = $(A-B) \times N \times 35.45 \times 1000$
- ml sample Where A = ml
- AgNO₃ required for sample
- B = ml AgNO₃
required for blank N
= Normality of

AgNO₃ used

Methods for Nitrate Estimation:

A) Ultraviolet spectrophotometric method

α. Useful for uncontaminated natural waters and potable water supplies that have low organic content

β. Follows Beer's law upto 11 mg/L as N

X. Interferences

- i. Dissolved Organic Matter**
- ii. Surfactants**
- iii. Nitrite and Hexavalent Chromium**

δ. Procedure

- i. Filter the sample
- ii. Add 1 ml of 1N HCl per 50 ml of sample
- iii. Read absorbance or transmittance at 220 nm and 275 nm

Set 0 absorbance or 100% transmittance with distilled water

Fluoride

Colorimetric SPADNS Method

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

Sulphate

Spectrophotometric Method

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

Calculate

$$\text{mg/L SO}_4 = \text{mgSO}_4 \times 1000$$

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 residual chlorine) (combined
- Ammonia concentration in water vary from less than 10ug 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 $\text{NH}_3\text{-N}$ concentrations more than $20\text{ }\mu\text{g/L}$
- 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 1cm light path
- A light path of 5 extends cm measurements of ammonia concentrations range of 5 to 60 µg/L

B) Ammonia Selective Electrode Method

- The ammonia selective electrode uses a hydro-phobic gas permeable membrane to separate the sample solution from an electrode internal solution of ammonium chloride
- Dissolved ammonia is converted to $\text{NH}_3(\text{aq})$ 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
- Applicable to the measurement of 0.03 to 1400 mg $\text{NH}_3\text{-N/L}$ 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

Phosphates

Methods For Phosphorous Estimation :

- 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 be removed by oxidation with bromine water.

Stannous Chloride Method

- Molybdophosphoric 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 be removed by oxidation with bromine water

• Procedure

❖ Sample pH adjustment if $\text{pH} > 10$

❖ 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
- 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.

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

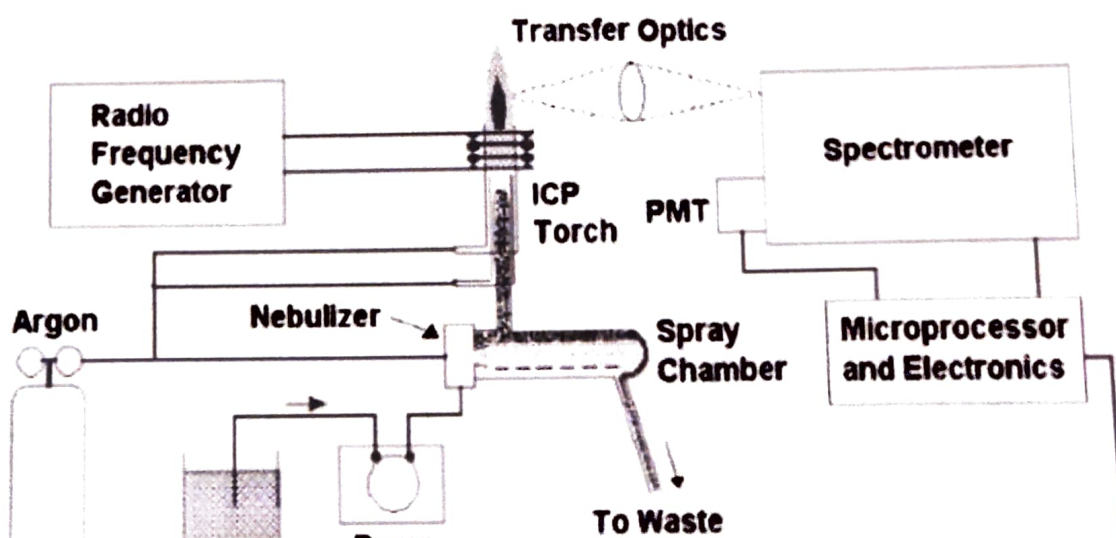
2. Manganese:- Persulphate method

Periodate method

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

Optima 4100 DV ICP-OES



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.

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 tungsten 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.

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 understood to be a measure of the capacity of water to precipitate 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

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 Gravimetric method

Total Hardness by Calculation:

$$\text{mg CaCO}_3/\text{L} = 2.497 [\text{Ca mg/L}] + 4.118 [\text{Mg mg/L}]$$

THANK YOU

Children with safe water



Thank You