# *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

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

# 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 (combined residual chlorine)

- Ammonia concentration in water vary from less than 10$\mu$g 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 $\mu$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 1cms light path

- A light path of 5 cm extends measurements of ammonia concentrations range of 5 to 60 $\mu$g/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 $1\mu g$ $NH_3$-N/ 50mL ($100\mu g$/L). However, reproducibility below 100 $\mu g$/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 $\mu$g 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