

C H A P T E R - II

MATERIALS AND METHODS

PHYSICO-CHEMICAL ANALYSIS

INTRODUCTION :

Physico chemical analysis is the prime consideration to assess the quality of water for its best usage say for drinking, bathing, fishing, industrial processing and so on, while for waste water either domestic or industrial to know the pollutional strength and its effect on the ecology.

River water often necessitates examination of water samples from different points and under varying conditions to find out the extent of pollution and natural purification that takes place in the water.

Well waters are examined to locate the potable sources of water as well as to study the effect of pumping in coastal areas, or in saline water tracts.

Waters are also examined to test the samples to ascertain their suitability for a particular tgrade, e.g. paper making, tanning, steam raising, dying, dairing etc. In such case a particular parameter assumes importance e.g. for steam raising water should be checked for hardness and dissolved oxygen, water used in textiles should be checked for iron and hardness.

Similarly domestic and industrial wastewaters are analysed for various parameters to decide upon what physical, chemical or biological treatment should be given to make them suitable for discharge either on land for irrigation or in other water bodies.

Comparatively this analysis for example determination of pH, temperature, DO, COD, TOC, can be done quick enough to adopt by regulatory agencies to monitor and control the ecological balance of nature.

ANALYTICAL PROCEDURE :

Quantitative analytical procedure fall into three broad groups, viz. gravimetric, volumetric and colorimetric estimations. Advanced techniques of analysis for certain parameters make use of special electrodes, atomic absorption spectrophotometer, chromatography etc. But these are classified as techniques for the micro-analytical procedures, and hence will be covered in a separate chapter.

i) Gravimetric Analysis : Gravimetric method depends on weighting solids obtained from the sample by evaporation, filtration or precipitation and generally used for macro analysis. These are time consuming procedures needing to drive away moisture before weighing. An analytical balance capable of weighing upto 0.0001 gm is necessary.

ii) Volumetric Analysis : Volumetric analysis depends on the measurement of volumes of liquid reagents of known strength. These need simple glasware like pipettes and burettes.

Indicators, standard solutions (solution of known-strength) have to be used. The strength of standard solutions are such that they are equivalent to one another. Dilute solutions of normality $N/10$, $N/50$, $N/80$, are used. It is convenient to adjust normalities to give the direct values of the substance/parameter to be determined from the volume of the titrant. Solutions used should be standardised before they are used for volumetric estimations. Standardisation is done with the help of primary standards which are salts or acid salts of high purity which can be accurately weighed. In volumetric analysis, various types of indicators are used to find out the end point. These are electrometric, acid base, precipitation, adsorption and oxidation-reduction.

iii) Colorimetric/Spectrophotometric Analysis : To be applicable quantitatively, a colorimetric/spectrophotometer method is based on formation of a compound or colour complex with definite colour characteristics. The coloured solutions must be homogeneous and obey -

- 1) Beer's law : The intensity of a beam of monochromatic light decreases exponentially as the concentration of the absorbing substance increases arithematically.
- 2) Lambert's Law : The intensity of the emitted light decreases exponentially as the thickness of the absorbing medium increases arithematically.

By combining the two absorption laws, the Beer Lambert law is obtained as -

$$T = \frac{I}{I_0} = 10^{-kcl}$$

$$A = \log \frac{I_0}{I} = \log \frac{I}{T} = kcl$$

where

T = fractional transmissin through sample.

A = Absorbance or optical density of the solution.

I_0 = Intensity of light entering solution.

I = Intensity of light leaving solution.

K = Constant for a particular solution.

C = Concentration of absorbing material in solution.

l = Length of the absorbing material in solution.

The colour produced can be measured visually with the help of Nessler's tubes or a colour comparator. These suffer from the drawback of being subject to personal judgement. The comparators are suitable only for field tests. Instrumental methods include use of a absorption meter or colorimeter or a spectrophotometer. These include a photoelectric cell coupled with a millivolt meter calibrated in terms of per cent transmission passes through the sample contained in neutral colourless glass cell. A suitable colour filter is introduced in the light path to give maximum light absorption by the sample. The filter is of the complementary colour to the colour of the

solution. This increases the sensitivity.

In a spectrophotometer, a prism or a grating is used to provide monochromatic light of the desired wavelength. While using a spectrophotometer, it is necessary to estimate the optimum wavelength for the colour developed in the sample. This wavelength gives the maximum absorption of light. Then at this wavelength a series of standard samples of known concentrations are used to obtain a calibration chart from which future sample readings are read off. It is necessary that full colour development takes place and turbidity is absent.

Types of Analysis :

For the sake of simplicity in understanding the parameters by their significance in physico-chemical analysis of water and waste water, they are categorised as i) Mineral Analysis consisting of physical parameters and significant anions and cations. (ii) Demand Analysis covering COD, TOC, BOD, D.O. permanganate value, etc. (iii) Nutrient Analysis consisting of different forms of nitrogen and phosphorous (iv) Heavy Metal Analysis covering analysis of heavy metals by different methods along with sample pretreatment.