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SUBJECT NAME: INSTRUMENTAL METHOD OF ANALYSIS

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CHAPTER 1: Nephelometry and Turbidimetry

Nephelometry and Turbidimetry

Nephelometry and Turbidimetry are used for continuous monitoring of air and water pollution. In water, turbidity is monitored whereas in air, smoke and dust are monitored. The techniques are also used in food, beverages and in the determination of molecular weight of high polymers which are settled down on earth from factories. The CO_2 can also be determined by this technique. Neptielommetric analysis is based on measuring the intensity of a luminous flux scattered by solid particles suspended in solution. Nephelommetric analysis (turbidimetry) is based on measuring the weakening of intensity of a luminous flux when h; passes through a solution containing particles in suspension. The intensity decreases owing to absorption and scattering of light.

Principle and Theory of Nephelometry and Turbidimetry:

The principle of nephelometry and turbidimetry is based on the scattering or absorption of light by solid or colloidal particles suspended in solution. When light is passed through the suspension, part of incident radiant energy is dissipated by absorption, reflection, and reaction while remainder is transmitted.

In fact the measurement of the intensity of transmitted light is a function of the concentration of dispersed phase and this becomes the basis of turbid metric analysis.**This is given in the following diagram**

$$\log \frac{I_0}{I_t} = k'lc. \qquad ...(1)$$

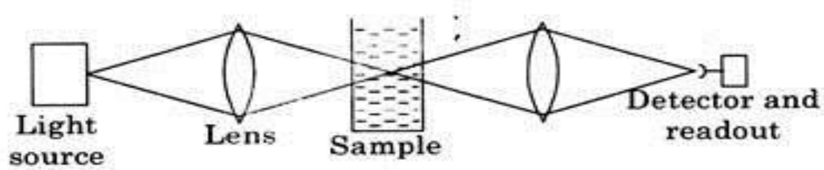

Fig. 5.

Nephelometry is somewhat different from turbidimetry. In nephelometry light is passed through the sample solution (suspended particles) directly and the amount of scattered radiation is measured generally at 90°C (Fig.6). The measurement of intensity of scattered light as a function of concentration of dispersed phase is the basis of analysis of nephelometry.

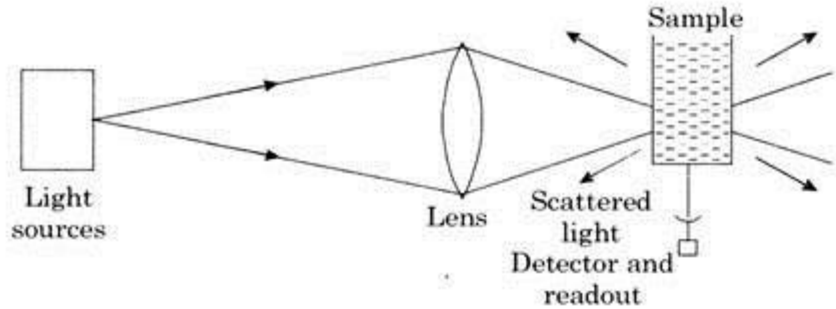

Fig. 6.

It is very important to note that in nephelometry incident and scattered light are of same wavelength whereas in fluorimeter (in fluorimetry) scattered light is of longer wavelength than incident light.

Theory:
For turbidmetric measurements the transmitted intensity I can be determined from the equation.

where I_0 = incident intensity.
I_t = transmitted intensity.
C = concentration of absorbing particles in the solution

1 = thickness of absorbing layer of solution.

This equation (1) is known as the basic equation of turbidimetry and is similar to Bouguer – Lambert – Beer equation:

$I_t = I_0 \times 10^{-k'lc}$
where k' is molar turbidity coefficient of solution.

Operating Conditions of Nephelometry and Turbidimetry:

In turbidimetric and nephelometric analysis a number of conditions must be satisfied for a successful working.

As the amount of light scattered or absorbed depends on size of the particles in the solution, hence correct results will depend upon method of preparing the suspensions and on reproducibility of their optical properties.

There are following factors which influence optical properties of suspension and particle size:
(a) Ratio of concentration of solutions mixed,

(b) Order of mixing of solutions,

(c) Concentration of ions forming the precipitate,

(d) Temperature,

(e) Rate of mixing,

(f) Presence of extraneous electrolytes,

(g) Stability of dispersion,

(h) Presence of non-electrolytes,

(i) Presence of protective colloids, and

(j) Time required to attain maximum turbidity.

Instrumentation of Nephelometry and Turbidimetry:

The instruments used in nephelometry and turbidimetry are similar as used in spectrophotometry.

We will describe here some special features:

(1) Sources:

Monochromatic radiation is used both in turbidimeters and nephelometer. Generally a mercury arc or a laser with special filter combinations for isolating one of its emission lines is the most suitable source.

The tungsten lamp (which is polychromatic source) is used when one has to determine the concentration of a particular substance. It has been observed that even in such a case blue spectral region gives the best results.

(2) Cells:

Generally a cell with a rectangular cross section is selected for the study. We can also use cylindrical cells having flat faces where entering and existing beams are passed. The octagonal faces allow measurements to be made at 135°C, 90° 45° or 0° to primary beam. The walls through which light beam are not to pass, are coated black so as to absorb unwanted radiation.

(3) Detectors:

In turbidimeters, phototubes are used as detectors.

The photo-mutliplier tubes are used as detectors in case of nephelometers because intensity of scattered radiation is generally very small. Generally the detector is fixed at 90° to primary but for maximum sensitivity the detector angle should vary. There are some nephelometers where detector is mounted on a circular disc which allows measurement at many angles, i.e. at 0° and from 35° to 135°.

Experimental Techniques of Nephelometry and Turbidimetry:

A powerful light from electric lamp passes through filter which is just put in place only where instrument is to be utilized for luminescence studies, and falls on glass plate. Some part of beam is reflected from this plate and falls on glass attenuator, while part of it enters cell filled with solution under study.

Now the light beam passing through cell is extinguished in light trap. The part of luminous flux reflected by particles in solution passes through lens, adjustable diaphragm, lens and that is directed by rhombic prism through filter into eye piece where it illuminates only one half of the optical field.

The luminous flux from attenuator traverses a similar path through lens, adjustable diaphragm, lens, rhombic prism, filter and enters eye piece to illuminate second half of the optical field. Now by varying slit width of adjustable diaphragms and luminous fluxes can be equalized, i.e. optical equilibrium is attained.

While working with this instrument solution under study should be placed in cell, dials of adjustable diaphragms are set to zero and intensities of luminous fluxes are brought close by putting in interchangeable attenuators. Then luminous fluxes are equalized by means of adjustable diaphragms.

Such measurements are made for a series of solutions containing definite quantities of substance analyzed and then a calibration curve is plotted relating the adjustable diaphragm readings to the concentration of solutions. After this, the unknown concentration can be determined from the diaphragm readings by using this calibration curve.

Calculation:

The concentration of metals or ions can be determined from the formula:

$C_1 = C_0\ 1_1/1_2$

where C_1 and C_2 are concentration of standard and unknown solution respectively.

1_1 and 1_2 are layer thickness in nephelometer cells.

Applications of Turbidimetry and Nephelometery:

Turbidimetry and nephelometry can be used in gaseous liquid or even transparent solid samples.

The applications or both these techniques can be summarized as follows:

(1) Air and Water Pollution:

Turbidimetry and nephelometry are used in the study of air and water pollution. In air, dust and smoke are measured whereas in water, turbidity is measured.

(2) Inorganic Analysis:

The main uses of nephelometry and turbidimetry are the determination of sulphate as $BaSO_4$, chloride as AgCl, fluoride as CaF_2, cyanide as AgCn, calcium as oxalate, carbonate as $BaCO_3$ and Zinc as ferrocyanide.

Nephlo-turbidimetric method no doubt is an ideal inexpensive technique used for multi-facet measurements some of which are precisely discussed below;

1. Determination of particle size present in suspensions.
2. Determination of average molecular weight of polymer in solution.
3. Measurement of atmospheric pollutants.
4. Determining concentration of solute in solution.
5. Growth of bacterial cell in a liquid nutrient medium.
6. Turbidimetry and nephelometry has numerous applications in water treatment plants, sewage work, steam generating plant, beverage bottling industry, in pulp and paper manufacturing, petroleum refining and pharmaceutical industries.
7. Determination of carbon dioxide, sugar products and clarity of citric acid juice.
8. Determining end point of precipitation titration.