Chapter Six
Instrumental Methods for monitoring Pollutants

6.1 Introduction

According to a more and more common opinion, analytics and monitoring of environmental pollutants constitute the two pillars on which all of environmental science is based. Consequently, one can share the opinion of some specialists that there already exists a separate field of chemical analytics called Eco analytics. However, we should be aware of the fact that neither analytics nor monitoring as such solves any problems concerning pollution or degradation of specific elements of the environment. They are only powerful tools which can provide information required for a reliable evaluation of the state of the environment and the changes taking place, as well as for making correct decisions for sozotechnical actions. The above tasks can be accomplished through the application of a wide range of procedures, analytical techniques and instruments. Monitoring should be considered as a specific branch of analytics where fully automated measuring devices are used. Requirements for this type of device are the following:

1. Methodical requirements: - high sensitivity of measurements, - producing analytical information continuously in real time or with only negligible delay, - high resolution of results characterized by short response time of the instruments, - longtime of autonomous operation.

2. Technical requirements: - automatic zeroing and instrument calibration, - protection against abrupt power failure, - equipping instruments with: - independent power supply, - calibration module, - system for filling and refilling solution and reagents (electronic monitoring of liquid level), - system protecting flames from extinguishing (monitors based on the use of FID and FPD detectors), - possibility of automatic regeneration or exchange of filters.

6.2 Conduct Metric Analyzer used for monitoring industrial pollutants:

This technique measures the conductance of an absorbing solution into which SO_2 from the sample has been dissolved by contact of the solution with the sample. As we know that an increase in conductance is caused by ions formed as SO_2 combines with the solution.

The two kinds of solutions used are demonized or distilled water and dilute acidified hydrogen peroxide solution. The observed increased in the conductivity is proportional to the SO_2concentration in the air if there are no interferences. So with the help of digital instrument, we can read directly the SO_2 concentration.

6.3 Atomic absorption spectroscopy (AAS)

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In analytical chemistry the technique is used for determining the concentration of a particular element (the analyze) in a sample to be analyzed. AAS can be used to determine over 70 different elements in solution or directly in solid samples used in pharmacology, biophysics and toxicology research.

Atomic absorption spectroscopy was first used as an analytical technique, and the underlying principles were established in the second half of the 19th century by Robert Wilhelm Bunsen and Gustav Robert Kirchhoff, both professors at the University of Heidelberg, Germany.

The modern form of AAS was largely developed during the 1950s by a team of Australian chemists. They were led by Sir Alan Walsh at the Commonwealth Scientific and Industrial Research

Organization (CSIRO), Division of Chemical Physics, in Melbourne, Australia.

Atomic absorption spectrometry has many uses in different areas of chemistry such as clinical analysis of metals in biological fluids and tissues such as whole blood, plasma, urine, saliva, brain tissue, liver, muscle tissue, semen, in some pharmaceutical manufacturing processes, minute quantities of a catalyst that remain in the final drug product, and analyzing water for its metal content.

Atomic absorption methods measure the amount of energy (in the form of photons of light, and thus a change in the wavelength) absorbed by the sample. Specifically, a detector measures the wavelengths of light transmitted by the sample (the "after" wavelengths), and compares them to the wavelengths, which originally passed through the sample (the "before" wavelengths). A signal processor then integrates the changes in wavelength, which appear in the readout as peaks of energy absorption at discrete wavelengths.

The process of atomic absorption spectroscopy (AAS) involves two steps:

1. Atomization of the sample
2. The absorption of radiation from a light source by the free atoms

The sample, either a liquid or a solid, is atomized in either a flame or a graphite furnace. Upon the absorption of ultraviolet or visible light, the free atoms undergo electronic transitions from the ground state to excited electronic states.

To obtain the best results in AA, the instrumental and chemical parameters of the system must be geared toward the production of neutral ground state atoms of the element of interest. A common method is to introduce a liquid sample into a flame. Upon introduction, the sample solution is dispersed into a fine spray, the spray is then desolated into salt

particles in the flame and the particles are subsequently vaporized into neutral atoms, ionic species and molecular species. All of these conversion processes occur in geometrically definable regions in the flame. It is therefore important to set the instrument parameters such that the light from the source (typically a hollow-cathode lamp) is directed through the region of the flame that contains the maximum number of neutral atoms. The light produced by the hollow-cathode lamp is emitted from excited atoms of the same element which is to be determined. Therefore the radiant energy corresponds directly to the wavelength which is absorbable by the atomized sample. This method provides both sensitivity and selectivity since other elements in the sample will not generally absorb the chosen wavelength and thus, will not interfere with the measurement. To reduce background interference, the wavelength of interest is isolated by a monochromatic placed between the sample and the detector.

6.4 Turbidimetry and Nephelometry:

When particles are suspended in a solution in a cuvette, they make the solution unclear (turbid). Incident light entering the cuvette will be subjected to three reactions;

1- Some of the light will be absorbed (blocked) by the particles

2- Some will be transmitted through the cuvette

3- Some will be scattered in various directions.

6.4.1 Turbidimetry

• Turbidimetry is involved with measuring the amount of transmitted light (and calculating the absorbed light) by particles in suspension to determine the concentration of the substance in question. Amount of absorbed light, and therefore, concentration is dependent on; a number of particles, and 2 size of particles.

• Measurements are made using light spectrophotometers Clinical Applications

• Determination of the concentration of total protein in biological fluids such as urine and CSF which contain small quantities of protein (mg/L quantities) using trichloroacetic acid

• Determination of amylase activity using starch as substrate. The decrease in turbidity is directly proportional to amylase activity.

6.4.2 Nephelometry.

• Nephelometry is concerned with measurement of scattered light from a cuvette containing suspended particles in a solution.

• The components of a nephelometer are the same as a light spectrophotometer except that the detector is placed at a specific angle from the incident light.

• The detector is a photomultiplier tube placed at a position to detect forward scattered light. Detectors may be placed at 90^o , 70^o or 37^o depending on the angle at which most scattered light are found.

• Since the amount of scattered light is far greater than the transmitted light in a turbid suspension, nephelometry offers higher sensitivity than turbidimetry.

• The amount of scattered light depends on the size and number of particles in suspension.

• For most clinical applications, the light source is a tungsten lamp giving light in the visible region

• For higher sensitivity and for applications that determine the size and number of particles in suspension, laser light nephelometers is used.

Considerations in turbidimetry and nephelometry

• The reaction in turbidimetry & nephelometry does not follow Beer's Law

• Therefore, standard curves must be plotted and the concentration of the unknown is determined from the standard curve.

• Because the absorbance is dependent on both number and size of particles, the standard solution which is used for the standard curve must have similar size in suspension as unknown.

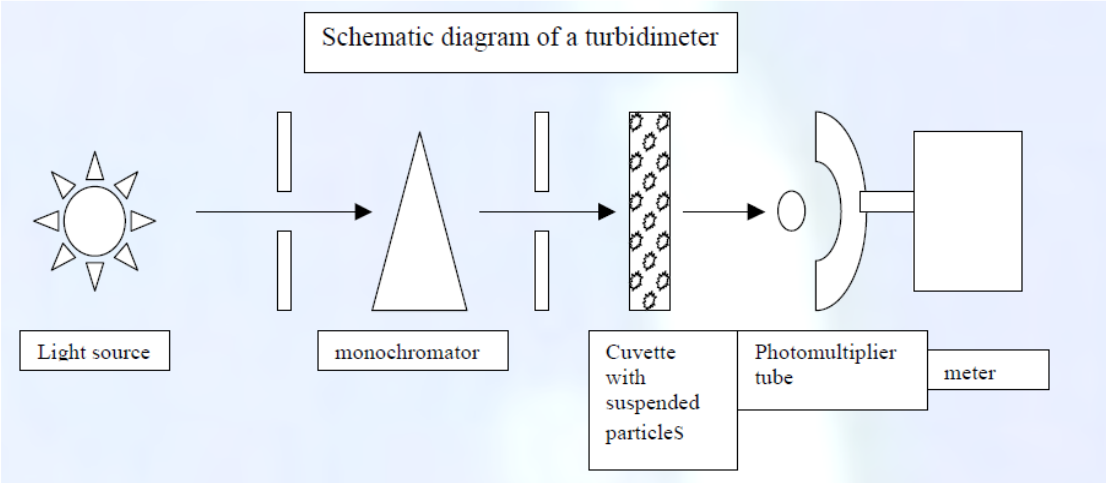
• Because some precipitation and settlement of particles may occur with time, in order to obtain good accuracy it is important to ; a) mix the

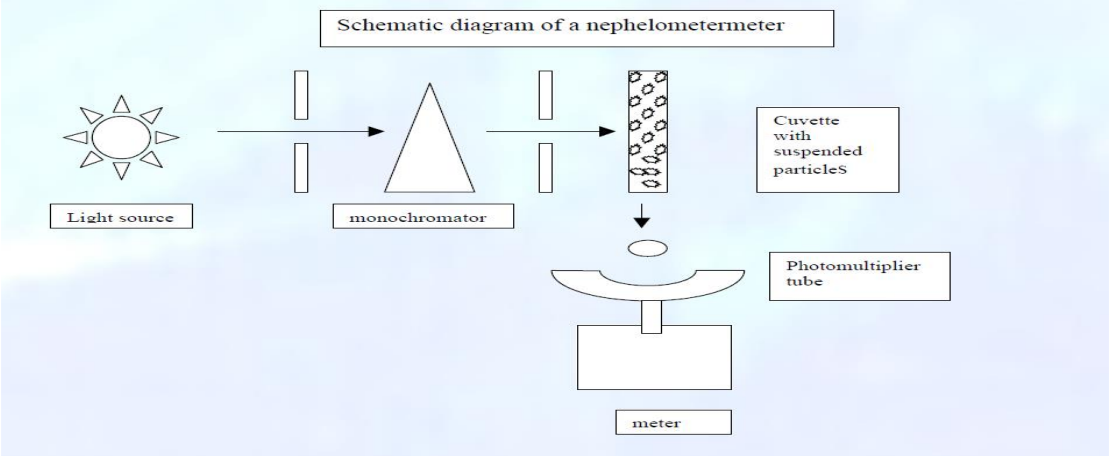
sample well prior to placing the cuvette in the instrument, and, b) keep the same time for measurement of every sample throughout the measurement.

• Kinetic reactions (measurement of the progress of reaction with time) provides higher degree of accuracy, sensitivity, precision and less time than end-point reactions (measuring the reaction at the start and finish of the reaction)

Additionally in kinetic reactions there is no need for reagent blank since the previous reading is taken as the base-line for the next reading.

Kinetic reaction may be taken in 60, 90 or 120 seconds (taking readings at 10 seconds intervals), whereas endpoint reactions may take much longer time e.g. 15 - 120 minutes.





6.5 Determination of disinfectants:

Disinfectants used in hospitals and laboratories must be tested periodically to ascertain its potency and efficacy. As certain disinfectants lose potency on standing and addition of organic matter, their efficacy must be tested. While certain methods help in selecting the right dilution of disinfectant for use others test the efficacy of disinfectant already in use. Some methods compare the performance with that of phenol whereas other methods simply state if the disinfectant is effective or not. There are several methods of testing disinfectants, with their own advantages and disadvantages. All these tests can be allocated to one of the following disinfectant tests: carrier test, suspension test, capacity test, practical test, field test or in-use test. **Disinfection process** validation is defined as "establishing documented evidence that a disinfection process will consistently remove or inactivate known or possible pathogens from inanimate objects."

6.5.1 In-use test:

A simple to use test was described by Maurer in 1985 that can be used in hospitals and laboratories to detect contamination of disinfectants. A 1 ml sample of the disinfectant is added to 9 ml diluent which also contains an inactivator. Ten drops, each of 0.02 ml volume of the diluted sample are

placed on each of two nutrient agar plates. One is incubated at 37o C for three days and the other at room temperature for seven days. Five or more colonies on either plate indicate contamination.

6.5.2 Bactericidal tests:

A bactericidal test must include the following sequence of steps:

1. The test organism is exposed to a suitable concentration of the disinfectant

2. Samples are taken at specified times and added immediately to a diluent or culture medium containing the appropriate disinfectant inactivator

3. The treated samples are cultured for surviving microorganisms.