Environmental Engineering Laboratory Manual

Semester - 5

Civil Engineering Department
Shantilal Shah Engineering College
Bhavnagar

Preface

This manual is prepared keeping in view the syllabus of the subject Environmental Engineering of 5th semester, Civil Engineering. The experiments and methods describe in this manual is as per the Standard Methods of APHA (American Public Health Association) for analysis of water and waste water. Guidelines suggested by CPCB (central pollution control board) are also incorporated while preparing this manual.

We hope this manual will be very helpful for the students to understand importance of various parameters and to perform analysis of water samples.

.

Civil Engineering Department
Shantilal Shah Engineering College,
Bhavnagar.

Gautam Dihora
Assistant Professor,
Civil Engineering Department

Table of Contents

1. Introduction to Equipment in Environmental Engineering Laboratory

Sr. no.	Name of equipment
1	Waterbath
2	Electornic Weight Balance
3	BOD Incubator
4	Water Quality Analyser
5	Hot air oven
6	Digital pH meter
7	Conductivity meter
8	Double Beam UV Visible Spectrophotometer
9	Autoclave
10	Noise lever meter
11	Exhaust Gas Analyser
12	TDS meter
13	Respirable dust sampler
14	Magnetic Stirrer
15	COD Digestor
16	Digital DO Meter
17	Jar Test Apparatus
18	Muffle Furnace
19	Colony counter
20	Colorimeter
21	Vaccume Pump
22	Top loading Balance
23	Nephelometric Turbidity Meter
24	Microscope
25	Bomb Calorimeter
26	Distillation Apparatus

2. Introduction to Standard, Sampling, Collection and Preservation of samples

Drinking Water Standards of BIS (IS: 10500:2012)

Sr. No.	Parameters	Desirable limit (mg/l)	Permissible limit (mg/l)
Organoleptic and Physical Parameters			
01	Colour Hazen unit, *Max*	5	15
02	Odour	Agreeable	
03	Taste	Agreeable	
04	Turbidity (NTU*), *Max*	1	5
05	pH	6.5 – 8.5	No relaxation
06	Total Dissolved Solids, *Max*	500	2000
General Parameters Concerning Substances Undesirable in Excessive Amounts			
01	Aluminium (as Al), *Max*	0.03	0.2
02	Ammonia (as total ammonia-N), *Max*	0.5	No relaxation
03	Anionic detergents (as MBAS), *Max*	0.2	1.0
04	Barium (as Ba), M*ax*	0.7	No relaxation
05	Boron (as B), *Max*	0.5	1.0
06	Calcium (as Ca), *Max*	75	200
07	Chloramines (as Cl_2), *Max*	4.0	No relaxation
08	Chloride (as Cl), *Max*	250	1000
09	Copper (as Cu), *Max*	0.05	1.5
10	Fluoride (as F), *Max*	1.0	1.5
11	Free residual chlorine, *Min*	0.2	1
12	Iron (as Fe), *Max*	0.3	No relaxation
13	Magnesium (as Mg), *Max*	30	100
14	Manganese (as Mn), *Max*	0.1	0.3
15	Mineral oil, *Max*	0.5	No relaxation
16	Nitrate (as NO_3), *Max*	45	No relaxation
17	Phenolic compounds (as C_6H_5OH), *Max*	0.001	0.002
18	Selenium (as Se), *Max*	0.01	No relaxation

19	Silver (as Ag), *Max*	0.1	No relaxation
20	Sulphate (as SO_4), *Max*	200	400
21	Sulphide (as H_2S), *Max*	0.05	No relaxation
22	Total alkalinity (as $CaCO_3$), *Max*	200	600
23	Total hardness (as $CaCO_{3)}$, *Max*	200	600
24	Zinc (as Zn), *Max*	5	15
Bacteriological Quality of Drinking Water			
01	All water intended for drinking a) E. coli or thermotolerant coliform bacteria	Shall not be detectable in any 100 ml sample	
02	Treated water entering the distribution system a) E. Coli or themotolerant coliform bacteria b) Total Coliform bacteria	Shall not be detectable in any 100 ml sample Shall not be detectable in any 100 ml sample	
03	Treated water in the distribution system a) E. Coli or themotolerant coliform bacteria b) Total Coliform bacteria	Shall not be detectable in any 100 ml sample Shall not be detectable in any 100 ml sample	

*NTU = Nephelometric Turbidity Unit

Collection and Preservation of Samples

The objective of sampling is to collect representative sample. Representative sample by means a sample in which relative proportions or concentration of all pertinent components will be the same as in the material being sampled. Moreover, the same sample will be handled in such a way that no significant changes in composition occur before the tests are made. The sample volume shall small enough that it can be transported and large enough for analytical purposes.

Because of the increasing placed on verifying the accuracy and representatives of data, greater emphasis is placed on proper sample collection, tracking, and preservation techniques

This section addresses the collection and preservation of water and wastewater samples; the general principles also apply to the sampling of solids or semisolid matrices.

Collection of Samples

Types of Samples

a. Grab samples:

Grab samples are single collected at a specific spot at a site over a short period of time (typically seconds or minutes). Thus, they represent a "snapshot" in both space and time of a sampling area. Discrete grab samples are taken at a selected location, depth, and time. Depth-integrated grab samples are collected over a predetermined part of the entire depth of a water column, at a selected location and time in a given body of water.

A sample can represent only the composition of its source at the time and place of collection. However, when a source is known to be relatively constant in composition over an extended time or over substantial distances in all directions, then the sample may represent a longer time period and/or a larger volume than the specific time and place at which it was collected. In such circumstances, a source may be represented adequately by single grab samples. Examples are protected groundwater supplies, water supplies receiving conventional treatment, some well-mixed surface waters, but rarely, wastewater streams, rivers, large lakes, shorelines, estuaries, and groundwater plumes.

When a source is known to vary with time, grab samples collected at suitable intervals and analyzed separately can document the extent, frequency, and duration of these variations. Choose sampling intervals on the basis of the expected frequency of changes, which may vary from as little as 5 min to as long as 1h or more. Seasonal variations in natural systems may necessitate sampling over months. When the source composition varies in space (i.e. from location to location) rather than time, collect samples from appropriate locations that will meet the objectives of the study (for example, upstream and downstream from a point source, etc.).

b. Composite Sample

Composite samples should provide a more representative sampling of heterogeneous matrices in which the concentration of the analytes of interest may vary over short periods of time and/or space. Composite samples can be obtained by combining portions of multiple grab samples or by using specially designed automatic sampling devices. Sequential (time)

composite samples are collected by using continuous, constant sample pumping or by mixing equal water volumes collected at regular time intervals. Flow-proportional composites are collected by continuous pumping at a rate proportional to the flow, by mixing equal volumes of water collected at time intervals that are inversely proportional to the volume of flow, or by mixing volumes of water proportional to the flow collected during or at regular time intervals.

Advantages of composite samples include reduced costs of analyzing a large number of samples, more representative samples of heterogeneous matrices, and larger sample sizes when amounts of test samples are limited. Disadvantages of composite samples include loss of analyte relationships in individual samples, potential dilution of analytes below detection levels, increased potential analytical interferences, and increased possibility of analyte interactions. In addition, use of composite samples may reduce the number of samples analyzed below the required statistical need for specified data quality objectives or project-specific objectives.

Do not use composite samples with components or characteristics subject to significant and unavoidable changes during storage. Analyze individual samples as soon as possible after collection and preferably at the sampling point. Examples are dissolved gases, residual chlorine, soluble sulfide, temperature, and pH. Changes in components such as dissolved oxygen or carbon dioxide, pH, or temperature may produce secondary changes in certain inorganic constituents such as iron, manganese, alkalinity, or hardness. Some organic analytes also may be changed by changes in the foregoing components. Use time-composite samples only for determining components that can be demonstrated to remain unchanged under the conditions of sample collection, preservation, and storage.

Collect individual portions in a wide-mouth bottle every hour (in some cases every half hour or even every 5 min) and mix at the end of the sampling period or combine in a single bottle as collected. If preservatives are used, add them to the sample bottle initially so that all portions of the composite are preserved as soon as collected.

Automatic sampling devices are available; however, do not use them unless the sample is preserved as described below. Composite samplers running for extended periods (week to months) should undergo routine cleaning of containers and sample lines to minimize sample growth and deposits.

c. Integrated (discharge-weighted) samples

For certain purposes, the information needed is best provided by analyzing mixtures of grab samples collected from different points simultaneously, or as nearly so as possible, using discharge-weighted methods such as equal-width increment (EWI) or equal discharge-increment (EDI) procedures and equipment. An example of the need for integrated sampling occurs in a river or stream that varies in composition across its width and depth. To evaluate average composition or total loading, use a mixture of samples representing various points in the cross-section, in proportion to their relative flows. The need for integrated samples also may exist if combined treatment is proposed for several separate wastewater streams, the interaction of which may have a significant effect on treatability or even on composition. Mathematical prediction of the interactions among chemical components may be inaccurate or impossible and testing a suitable integrated sample may provide useful information.

Both lakes and reservoirs show spatial variations of composition (depth and horizontal location). However, there are conditions under which neither total nor average results are especially useful, but local variations are more important. In such cases, examine samples separately (i.e., do not integrate them).

Preparation of integrated samples usually requires equipment designed to collect as ample water uniformly across the depth profile. Knowledge of the volume, movement, and composition of the various parts of the water being sampled usually is required. Collecting integrated samples is a complicated and specialized process that must be described in a sampling plan.

2.1.1. General Requirement

- Obtain a sample that meets the requirements of the sampling program and handle it so that it does not deteriorate or become contaminated before it is analyzed.
- Ensure that all sampling equipment is clean and quality-assured before use. Use sample containers that are clean and free of contaminants. Bake at 450°C all bottles to be used for organic analysis sampling.
- Fill sample containers without prerinsing with sample; prerinsing results in loss of any pre-added preservative and sometimes can bias results high when certain components adhere to the sides of the container. Depending on determinations to be performed, fill the container full (most organic compound determinations) or leave space for aeration, mixing, etc. (microbiological and inorganic analyses). If the bottle already contains

preservative, take care not to overfill the bottle, as preservative may be lost or diluted. Except when sampling for analysis of volatile organic compounds, leave an air space equivalent to approximately 1% of the container volume to allow for thermal expansion during shipment.

- Special precautions (discussed below) are necessary for samples containing organic compounds and trace metals. Since many constituents may be present at low concentrations (micro-grams or nanograms per liter), they may be totally or partially lost or easily contaminated when proper sampling and preservation procedures are not followed.
- Composite samples can be obtained by collecting over a period of time, or at many different over a period of time, depth, or at many different sampling points. The details of collection vary with local conditions, so specific recommendations are not universally applicable. Sometimes it is more informative to analyze numerous separate samples instead of one composite so that variability, maxima, and minima can be determined.
- Because of the inherent instability of certain properties and compounds, composite sampling for some analytes is not recommended where quantitative values are desired (examples in-residual, iodine, hexavalent chromium, nitrate, volatile organic compounds, radon-222, dissolved oxygen, ozone, temperature, and pH). In certain cases, such as for BOD, composite samples are routinely by regulatory agencies. Refrigerate composite samples for BOD and nitrite.
- Important factors affecting results are the presence of suspended matter or turbidity, the method chosen for removing a sample from its container, and the physical and chemical brought about by storage or aeration. Detailed procedures are essential when processing (blending, sieving, filtering) samples to be analyzed for trace constituents, especially metals and organic compounds. Some determinations can be invalidated by contamination during processing. Treat each sample individually with regard to the substances to be determined, the amount and nature of turbidity present, and other conditions that may influence the results.
- For metals it often is appropriate to collect both a filtered and an unfiltered sample to differentiate between total and dissolved metals present in the matrix. Be aware that some metals may partially sorb to filters. Beforehand, determine the acid requirements to bring the pH to <2 on a separate sample. Add the same relative amount of acid to all

samples; use ultrapure acid preservative to prevent contamination. When filtered samples are to be collected, filter them, if possible, in the field, or at the point of collection before preservation with acid. Filter samples in a laboratory-controlled environment if field conditions could cause error or contamination; in this case filter as soon as possible. Often slight turbidity can be tolerated if experience shows that it will cause no interference in gravimetric or volumetric tests and that its influence can be corrected in colorimetric tests, where it has potentially the greatest interfering effect. Sample collector must state whether or not the sample has been filtered.

- Record of sample shall be as follows:

General information

- Sample identification number
- Location
- Sample collector
- Date and hour
- Sample type (Grab or composite)

Specific information

- Water temperature
- Weather
- Stream blow
- Water level
- Any other information

The information may be attached tag, labeling or writing on container with water proof ink.

Description of sampling points

- By map using landmarks
- Use global positioning system

2.1.2. Sample Storage and Preservation

Complete and unequivocal preservation of samples, whether domestic wastewater, industrial wastes, or natural waters, is a practical impossibility because complete stability for every constituent never can be achieved. At best, preservation techniques only retard chemical and biological changes that inevitably continue after sample collection.

2.1.2.1. Sample Storage before Analysis

a. Nature of sample changes:

Some determinations are more affected by sample storage than others. Certain cations are subject to loss by adsorption on, or ion exchange with, the walls of glass containers. These include aluminium, cadmium, chromium, copper, iron, lead, manganese, silver and zinc, which are best collected in a separate clean bottle and acidified with nitric acid to a pH below 2.0 to minimize precipitation and adsorption on container walls. Also, some organics may be subject to loss by adsorption to the walls of glass containers.

Temperature changes quickly; pH may change significantly in a matter of minutes; dissolved gases (oxygen, carbon dioxide) may be lost. Because in such basic conductance, turbidity, and alkalinity immediately after sample collection. Many organic compounds are sensitive to changes in pH and/or temperature resulting in reduced concentrations during storage.

Changes in the pH-alkalinity-carbon dioxide balance may cause calcium carbonate to precipitate, decreasing the values for calcium and total hardness.

b. Time interval between and analysis:

In general, the shorter the time that elapses between collection of a sample and its analysis, the more reliable will be the analytical results. For certain constituents and physical values, immediate analysis in the field is required. For composited samples it is common practice to use the time at the end of composite collection as the sample collection time.

2.1.2.2. Preservation Techniques

To minimize the potential for volatilization or biodegradation between sampling and analysis, keep samples as cool as possible without freezing. Preferably pack samples in crushed or cubed ice or commercial ice substitutes before shipment. Avoid using dry ice because it will freeze samples and may cause glass containers to break. Dry ice also may effect a pH change in samples. Keep composite samples cool with ice or a refrigeration system set at 4ºC during compositing. Analyze samples as quickly as possible on arrival at the laboratory. If immediate analysis is not possible, preferably store at 4°C

No single method of preservation is entirely satisfactory; choose the preservative with due regard to the determinations to be made. Use chemical preservatives only when they do not interfere with the analysis being made. When they are used, add them to the sample bottle initially so that all sample portions are preserved as soon as collected. Because a preservation method for one determination may interfere with another one, samples for multiple

determinations may need to be split and preserved separately. All methods of preservation may be inadequate when applied to suspended matter. Do not use formaldehyde as a preservative for samples collected for chemical analysis because it affects many of the target analytes.

Methods of preservation are relatively limited and are intended generally to retard biological action, retard hydrolysis of constituents.

3. Presumptive test for coliform bacteria

The Multiple Tube Test

The following tests determine if coliform organisms are present and are much more significant than the plate count in determining if fecal contamination has occurred. The presence of these organisms may be an indication that harmful bacteria are entering the water supply.

The presumptive test:

Coliform bacteria are grown in test tubes containing Lactose Broth in which a water sample is placed. The Lactose Broth provides the moisture needed for growth, and few other organisms grown in the broth. The test tubes are placed in an incubator at a temperature of 98.6°F.

If food, moisture, and proper temperature are provided for the proper time, the organisms, if present, will grow. If coliform organisms or the few other types which will grow in Lactose are not present in the water, then no growth occurs. However, if organisms are present, they will grow under these ideal conditions and will ferment the Lactose Broth and produce gas. The gas indicates the presence of the organisms.

After 24 hours in the incubator, the tubes are examined for gas. If no gas has formed, they are given an additional 24 hours time. If, after 48 hours, gas has not formed, then no organisms were present and the report reads "Absent" and the water is considered safe for drinking purposes.

If organisms that reproduce in Lactose Broth are present in the sample, gas will be produced in any or all of the tubes within 18 hours. Because of the presence of gas, it is known that some type organism is present; and it is presumed that the organisms are coliform. That is the reason the test is called the "presumptive test", it is merely presumed that coliform organisms are present if gas is produced in Lactose Broth.

4. Determination of pH and conductivity for water and wastewater

4.1. pH

The pH of a solution is measured as negative logarithm of hydrogen ion concentration. At a given temperature, the intensity of the acidic or basic character of a solution is indicated by pH or hydrogen ion concentration. pH values from 0 to 7 are diminishing acidic, 7 to 14 increasingly alkaline and 7 is neutral.

Measurement of pH in one of the most important and frequently used tests, as every phase of water and wastewater treatment and waste quality management is pH dependent.

The pH of natural water usually lies in the range of 4 to 9 and mostly it is slightly basic because of the presence of bicarbonates and carbonates of alkali and alkaline earth metals. pH value is governed largely by the carbon dioxide/ bicarbonate/ carbonate equilibrium. It may be affected by humic substances, by changes in the carbonate equilibriums due to the bioactivity of plants and in some cases by hydrolysable salts. The effect of pH on the chemical and biological properties of liquid makes its determination very important. It is used in several calculations in analytical work and its adjustment to an appropriate value is absolutely necessary in many of analytical procedures.

In dilute solution, the hydrogen ion activity is approximately equal to the concentration of hydrogen ion. Pure water is very slightly ionized and at equilibrium the ionic product is:

$[H^+]\ [OH^-] = K = 1.0 \times 10^{-14}$ at 25°C

OR

$[H^+] = [OH^-] = 1.005 \times 10\text{-}7$

A logarithmic form is,

$[-\log_{10}(H^+)]\ [-\log_{10}(OH^-)]$

Or

$pH + pOH = pKw$

A. Electronic Method

From the above equilibrium it is clear that the pH scale for an aqueous solution lies between 0 and 14. The pH determination is usually done by electrometric method, which is the most accurate one, and free from interferences. The Colorimetric indicator methods can be used only if approximate pH values are required.

4.1.1. Principle

The pH is determined by measurement of the electromotive force (emf) of a cell comprising of an indicator electrode (an electrode responsive to hydrogen ions such as glass electrode) immersed in the test solution and a reference electrode (usually a calomel electrode). Contact is achieved by means of a liquid junction, which forms a part of the reference electrode. The emf of this cell is measured with pH meter.

Since the pH is defined operationally on a potentiometric scale, the measuring instrument is also calibrated potentiometrically with an indicating (glass) electrode and a reference electrode using standard buffers having assigned pH value so that

$pH_B = -\log10[H^+]$

Where pH_B = assigned pH of standard buffer.

The operational pH scale is used to measure sample pH and is defined as:

$pHs = pH_B + F (Es - E_B) / 2.303\ RT$

where,

pHs = potentiometrically measured sample pH

F = Faraday 9.649×10^4 coutomb/mole

Es= Sample emf V

E_B= Buffer emf V

R = Gas constant 1.987 cal deg-1 mole-1

T = absolute temperature, °K

4.1.2. Apparatus and equipment

a. pH meter: Consisting of potentiometer, a glass electrode, a reference electrode and a temperature compensating device. A balanced circuit is completed through potentiometer when the electrodes are immersed in the test solution. Many pH meters are capable of reading pH or millivolt.

b. Reference electrode: Consisting of a half cell that provides a standard electrode potential. Generally calomel, silver-silver chloride electrodes are used as reference electrode.

c. Sensor (glass) electrode: Several types of glass electrodes are available. The glass electrode consists essentially of a very thick walled glass bulb, made of low melting point glass of high electrical conductivity, blown at the end of a glass tube. This bulb contains an electrode, which has a constant potential, e.g. a

platinum wire inserted in a solution of H^+ hydrochloric acid saturated with quinhydrone. The bulb is placed in the liquid where pH is to be determined.

d. Beakers: Preferably use polyethylene or TFE beakers.

e. Stirrer: Use a magnetic TFE coated stirring bar.

4.1.3. Reagents and standards

a. pH 4 buffer solution: Dissolve 10.12g potassium hydrogen phthalate, $KHC_8H_4O_9$ in distilled water. Dilute to 1L.

b. pH 7 buffer solution: Dissolve 1.361g anhydrous potassium dihydrogen phosphate, KH_2PO_4, and 1.42g anhydrous disodium hydrogen phosphate, Na_2HPO_4, which have been dried at 110°C. Use distilled water which has been boiled and cooled. Dilute to 1L.

c. pH 9.2 buffer solution: Dissolve 3.81gm borax, $Na_2B_4O_7.10H_2O$ in distilled water, which has been previously boiled and cooled. Dilute to 1L.

4.1.4. Calibration

Before use, remove the electrodes from the water and rinse with distilled or demineralised water. Dry the electrodes by gentle wiping with a soft tissue. Calibrate the electrode system against standard buffer solution of known pH. Because buffer solution may deteriorate as a result of mould growth or contamination, prepare fresh as needed for work or use readily available pH buffers. Use distilled water a conductivity of less than2μ siemens at 25°C and distilled and pH 5.6 to 6.0 for the preparation of all standard solutions. For routine analysis, commercially available buffer tablets, powders or solutions of tested quality also are permissible. Buffer having pH 4.0, 7.0 and 9.2 are available. In preparing buffer solutions from solid salts, dissolve all the material; otherwise, the pH calibration will be incorrect. Prepare and calibrate the electrode system with buffer solutions with pH approximating that of the sample, to minimise error resulting from nonlinear response of the electrode.

4.1.5. Procedure

a. Before use, remove electrodes from storage solutions (recommended by manufacturer) and rinse with distilled water+

b. Dry electrodes by gently blotting with a soft tissue paper, standardise instrument with electrodes immersed in a buffer solution within 2 pH units of sample pH.

c. Remove electrodes from buffer, rinse thoroughly with distilled water and blot dry.

d. Immerse in a second buffer below pH 10, approximately 3 pH units different from the first, the reading should be within 0.1 unit for the pH of second buffer. (If the meter response shows a difference greater than 0.1 pH unit from expected value, look for trouble with the electrodes or pH meter)
e. For samples analysis, establish equilibrium between electrodes and sample by stirring sample to ensure homogeneity and measure pH.
f. For buffered samples (or those with high ionic strength), condition the electrodes after cleaning by dipping them into the same sample, and read pH.
g. With poorly buffered solutions (dilute), equilibrate electrodes by immersing in three or four successive portions of samples. Take a fresh sample and record the pH.

4.1.6. Calculation

The pH value is obtained directly from the instrument.

4.2. Conductivity

Conductivity is the capacity of water to carry an electrical current and varies both with number and types of ions in the solutions, which in turn is related to the concentration of ionized substances in the water. Most dissolved inorganic substances in water are in the ionized form and hence contribute to conductance.

4.2.1. Principle

This method is used to measure the conductance generated by various ions in the solution/water. Rough estimation of dissolved ionic contents of water sample can be made by multiplying specific conductance (in mS/cm) by an empirical factor which may vary from 0.55 to 0.90 depending on the soluble components of water and on the temperature of measurement.

Conductivity measurement gives rapid and practical estimate of the variations in the dissolved mineral contents of a water body.

4.2.2. Apparatus and equipment

a. Self-contained conductance instruments: (Conductivity meter). These are commercially available.
b. Thermometer, capable of being read to the nearest 0.1°C and covering the range 10-50°C.

c. Conductivity Cells: The cell choice will depend on the expected range of conductivity and the resistance range of the instrument. Experimentally check the range of the instruments assembly by comparing the instrumental results with the true conductance of the potassium chloride solution.

4.2.3. Reagents and standards

Conductivity Water: The conductivity of the water should be less than 1 mmho/cm; Standard potassium chloride: 0.01M; dissolve 745.6mg anhydrous KCl in conductivity water and make up to 1,000mL at 25°C. This is the standard reference solution, which at 25°C has a specific conductance of 1,413mmhos/cm. It is satisfactory for most waters when using a cell with a constant between 1 and 2. Store the solutions in glass stoppered Pyrex bottles.

4.2.4. Procedure

Conductivity can be measured as per the instruction manual supplied with the instrument and the results may be expressed as mS/m or mS/cm. Note the temperature at which measurement is made. Conductivity meter needs very little maintenance and gives accurate results. However few important points in this respect are:

a. Adherent coating formation of the sample substances on the electrodes should be avoided which requires thorough washing of cell with distilled water at the end of each measurement.
b. Keep the electrode immersed in distilled water
c. Organic material coating can be removed with alcohol or acetone followed by washing with distilled water.

4.2.5. Calculation

Follow the instruction manual.

5. Determination of Solids (Suspended, dissolved and settleable)

5.1. Introduction:

All matter except the water contained in liquid materials classified as “solid waste”. The usual definition of solids, however refers to the matter that remain as residue upon evaporation and drying at 103°C to 105°C.

5.2. Objective:

Determination of suspended, dissolved and settleable solids.

5.3. Principle:

Concentration of solids in water depends upon the source of water and the soil strata from which the water is percolating and starts dissolving the soluble salts. Total solid is the term applied to the residue left in the vessel after evaporation of the sample and its drying in the oven at a definite temperature about 103°C to 105°C.

The total solid enclose total suspended solids, is that portion of total solid remain on the filter paper and the total dissolved solid, is that portion which passes through the filter. Dissolved solids are the portion of solids that passes through normal general size of 2mμ under specified conditions and bigger than that will be retained as suspended solids on the filter paper.

5.4. Apparatus and equipment

a. Electrically heated temperature controlled oven
b. Analytical balance
c. Steam bath
d. Evaporating dish-Porcelain (200ml)
e. Pipettes
f. Desiccator
g. Measuring cylinder (100ml)

5.5. Procedure:

5.5.1. Determination of Total solids and dissolved solids

1. Take an evaporating dish and clean it properly to remove all the impurities.
2. Dry it to 103°C in an oven for 1hr and weigh (W1). Weighing should be carried out after transferring the evaporating dish in the desiccator.

3. Take 50 ml of water sample and transfer it in an evaporating dish.
4. Put it on a steam bath and allow the sample to evaporate
5. After complete evaporating, dry the evaporating dish with residue in oven at 103°C for 1 hr
6. Cool the evaporating dish in desiccator and take weight (W2)
7. Take another 50 ml sample and filter it on filter paper to remove suspended solids.
8. Collect the filtrate in evaporating dish
9. Put it on a steam bath and allow the sample to evaporate
10. Dry the evaporating dish in oven at 180°C for 1 hr.
11. Cool the evaporating dish in desiccator and take weight (W4)

Observation:

1. Weight of empty evaporating dish (dried at 103°C) (W1) =
2. Weight of evaporating dish + residue of sample dried at 103°C (W2) =
3. Weight of empty evaporating dish (dried at 103°C) (W3) =
4. Weight of evaporating dish + residue of filtered sample dried at 180°C (W4) =

Calculation:

Total solids, mg/l A $= \frac{(W2 - W1) \times 1000}{ml\ of\ sample}$

Dissolved solids, mg/l B $= \frac{(W4 - W3) \times 1000}{ml\ of\ sample}$

Suspended Solids, mg/l C $= A - B$

5.5.2. Determination of Settleable solids

1. Determine total suspended solids of sample as describe above.
2. Take 1 L sample and pour well mixed sample into a glass vessel of greater diameter

3. Let stand quiescent for 1h and without disturbing the settled or floating material, siphon about 250 ml from center of the container at a point halfway between the surface of the settled material and the liquid surface
4. Determine total suspended solids of this supernatant liquor. These are the non settleable solids.

Observation:

1. Weight of empty evaporating dish (dried at 103°C) (W5) =
2. Weight of evaporating dish + residue of sample dried at 103°C (W6) =

Calculation:

Total solids (non settleable), mg/l D = $\frac{(W6 - W5) \times 1000}{ml\ of\ sample}$

Settleable Solids, mg/l = C – D

6. Determination of Acidity, Alkalinity and Hardness

6.1. Acidity

Acids contribute to corrosiveness and influence chemical reaction rates, chemical speciation and biological processes. Acidity of water is its quantitative capacity to react with a strong base to a designated pH. The measured value may vary significantly with the end point pH used in the determination. When the chemical composition of the sample is known study mineral acids, weak acids such as carbonic and acetic and hydrolyzing salts such as iron or aluminum sulfate may contribute to the measured acidity according to the method of determination.

Mineral acidity: It is measured by titration to a pH of about 3.5, the methyl orange end point (also known as methyl orange acidity). Total acidity: Titration of a sample to the phenolphthalein end point of pH 8.3 measures mineral acidity plus acidity due to weak acids, thus this is called as total acidity (or phenolphthalein acidity). In water analysis, this test does not bear significant importance because methyl orange acidity invariably remains absent in the raw water and even phenolphthalein acidity (that too principally due to the excessive-prevalence of dissolved carbon dioxide and carbonic acids) normally does not exist to a significant extent in the raw water.

6.1.1. Apparatus

a. pH meter

6.1.2. Reagents

a. Sodium hydroxide titrant (0.02N)
b. Phenolphthalein indicator
c. Methyl orange indicator

6.1.3. Procedure

1 Take 50 ml sample in a conical flask and add 2-3 drops of methyl orange indicator solution

2 Fill the burette with 0.02 N NaOH solution and titrate till the colour of solution just changes to faint orange colour, indicating the end point. Record the volume of titrant consumed as V1 in ml. Calculate the methyl orange acidity using following equation

Methyl orange acidity (or Mineral acidity) = (v1 × 1000)/(sample volume)

When the 0.02 N NaOH solution, used in titration is not standardized, mineral acidity is calculated using following equation

Methyl orange acidity = (V1 × N × 50 × 1000)/(sample volume)

3 For phenolphthalein acidity test, add 2-3 drops of phenolphthalein indicator solution to water sample from step 2 and continue the titration till the faint pink colour develops in the solution (i.e., the end point of titration). Record the volume of titration consumed as V2 (mL) and calculate total acidity or phenolphthalein acidity using following equation

Total acidity (or phenolphthalein acidity) = (V2 × N × 50 × 1000)/(sample volume)

6.2. Alkalinity

6.2.1. Principle

Alkalinity of sample can be estimated by titrating with standard sulphuric acid (0.02N) at room temperature using phenolphthalein and methyl orange indicator. Titration to decolourisation of phenolphthalein indicator will indicate complete neutralization of OH^- and ½ of CO_3^{--}, while sharp change from yellow to orange of methyl orange indicator will indicate total alkalinity (complete neutralisation of OH^-, CO_3^{--}, HCO_3^-).

6.2.2. Apparatus

a. Beakers
b. Pipettes (Volumetric)
c. Flasks (Volumetric)

6.2.3. Reagents and Standards

a. Standard H2SO4, 0.02 N:
b. Phenolphthalein indicator
c. Methyl orange indicator

6.2.4. Calibration

Standardise the pH meter by using pH buffers. Follow the instructions given in the manual of pH meter.

6.2.5. Procedure

a. Take 25 or 50mL sample in a conical flask and add 2-3 drops of phenolphthalein indicator.
b. If pink colour develops titrate with 0.02N H_2SO_4 till disappears or pH is 8.3. Note the volume of H_2SO_4 required.
c. Add 2-3 drops of methyl orange to the same flask, and continue titration till yellow colour changes to orange. Note the volumes of H_2SO_4 required.
d. In case pink colour does not appear after addition of phenolphthalein continue as above.
e. Alternatively, perform potentiometric titration to preselected pH using appropriate volume of sample and titration assembly. Titrate to the end point pH without recording intermediate pH.

   As the end point is approached make smaller additions of acid and be sure that pH equilibrium is reached before adding more titrant. The following pH values are suggested as equivalence points for corresponding alkalinity as mg $CaCO_3$/L

End point pH values

Alkalinity range and Nature of sample	End point pH	
	Total Alkalinity	Phenolphthalein Alkalinity
Alkalinity, mg $CaCO_3$/L 30 150 500	 4.9 4.6 4.3	 8.3 8.3 8.3
Silicates, phosphates known or suspended	4.5	8.3
Routine or automated analyses	4.5	8.3
Industrial waste or complex system	4.5	8.3

6.2.6. Calculation

Calculate total (T), phenolphthalein (P) alkalinity as follows:

P-alkalinity, as mg $CaCO_3$/L = A x 1000/mL sample

T-alkalinity, as mg $CaCO_3$/L = B x 1000/mL sample

In case H_2SO_4 is not 0.02 N apply the following formula:

Alkalinity, as mg $CaCO_3$/L = A/B x N x 50000 / mL of sample

Where,

A = mL of H_2SO_4 required to bring the pH to 8.3

B = mL of H_2SO_4 required to bring the pH to 4.5

N = normality of H_2SO_4

Once, the phenolphthalein and total alkalinities are determined, three types of alkalinities, i.e. hydroxide, carbonate and bicarbonate are easily calculated from the table given as under:

Type of alkalinity

Values of P and T	Type of Alkalinity		
	Hydroxide Alkalinity as $CaCO_3$	Carbonate Alkalinity as $CaCO_3$	Bicarbonate Alkalinity as $CaCO_3$
P = 0	0	0	T
P < 1/2T	0	2P	T – 2P
P = 1/2T	0	2P	0
P > 1/2T	2P – T	2(T – P)	0
P = T	T	0	0

Once carbonate and bicarbonate alkalinities are known, then their conversions to milligrams CO_3^{--}or HCO_3^{-}/L are possible.

mg CO_3^{--}/L = Carbonate alkalinity mg $CaCO_3$/L x 0.6

mg HCO_3 = Bicarbonate alkalinity mg $CaCO_3$/L x 1.22

from above, molar concentration may be obtained as follows:

$[CO_3^{--}]$ = mg/L CO_3 / 60000

$[HCO_3^{-}]$ = mg/L HCO_3^{-}/ 61000

6.3. Hardness

Water hardness is a traditional measure of the capacity of water to precipitate soap. Hardness of water is not a specific constituent but is a variable and complex mixture of cations and anions. It is caused by dissolved polyvalent metallic ions. In fresh water, the principal hardness causing ions are calcium and magnesium which precipitate soap. Other polyvalent cations also may precipitate soap, but often are in complex form, frequently with organic constituents, and their role in water hardness may be minimal and difficult to define. Total hardness is defined as the sum of the calcium and magnesium concentration, both expressed as $CaCO_3$, in mg/L. The degree of hardness of drinking water has been classified in terms of the equivalent $CaCO_3$ concentration as follows:

Soft 0 - 75 mg/L

Medium 75 - 150 mg/L

Hard 150 – 300 mg/L

Very hard >300 mg/L

6.3.1. Principle

Ethiline diemine tetra acitate (EDTA) and its sodium salts forms a geletanious soluble complex when added to a solution of certain metallic cations. If a some amount of chrome black T is added to sample at pH = 10, it will give wine red colour with calcium and magnesium salts. If EDTA is added, it reacts with calcium and magnesium from dry complex. The solution will turn from wine red to blue colour. This is the end point of titration. Interferences some metalions interfere with this procedure by causing indistinct end point. This interference is reduce by addition of inhabitance to the water sample before titration with EDTA.

6.3.2. Reagents:

1. Standard EDTA Solution

   Dissolve 3.732 gms of sodium salts of EDTA in distilled water and dilute it to 1 liter by distilled water. This will produce a standard EDTA solution having strength of 1 ml of EDTA solution = 1 mg of $CaCO_3$.

2. Buffer Solution

   Dissolve 16.9 gms of ammonium chloride (NH_4Cl) in 143 ml concentrated ammonium hydroxide (liquor ammonia). Add 1.25 gms sodium salts of EDTA and dilute to 250ml with distilled water.

3. Inhibitor solution

   Dissolve 5 gm of $Na_2S.9H_2O$ or 3.7 gms of $Na_2S.5H_2O$ in 100 ml of distilled water.

4. Indicator solution (Chrome black T)

   Dissolve 0.5 gms of chrome black T in 100 ml of 95% Ehile alcohol at 4.5 gms of hydroximine hydrochloride ($NH_2OH.HCl$).

6.3.3. Procedure:

1. Take 20 ml or suitable amount of sample and dilute to 50 ml with distilled water
2. Add 1 ml of buffer solution followed by 1 ml of inhibitor.
3. Mix well and add 2 drops of indicator solution (Chrome black T)
4. Add the titrant (EDTA) with continuous stirring until the solution become blue from wine red colour.

6.3.4. Calculation for Total Hardness

$$\text{Total Hardness} = \frac{\text{ml of titrant (EDTA) used} \times 1000}{\text{ml of Sample taken}}$$

6.3.5. Determination of Carbonate Hardness

Alkalinity of water is usually imparted by carbonate, bicarbonate and hydroxide components of water. Carbonate hardness is equivalent to the total alkalinity of water as it is determine by titration with standard sulphuric acid reagent

6.3.6. Reagent

1. Standard sulphuric Acid (0.02 N)

   Take 3 ml of 36N concentrated H_2SO_4 and dilute it to 1 liter, this gives 0.1N H_2SO_4. Take 200 ml of this solution and dilute further to 1 liter to get 0.02 N H_2SO_4.
2. Methyl Orange Indicator

   Dissolve 0.5 gms methyl orange in 1 liter distilled water.

6.3.7. Procedure:

1. Take 20 ml of sample or any suitable amount of sample and dilute it to 50 ml by distilled water.
2. Transfer it to flask and add 2 drops of methyl orange indicator. The solution forms yellow colour.
3. Titrate it with 0.2N H_2SO_4 till colour changes from yellow to pinkish. This is the end point of titration.
4. Note down the amount of titrant (0.2N H_2SO_4) used in titration.

6.3.8. Calculation for carbonate hardness:

$$\text{Carbonate Hardness} = \frac{\text{ml of titrant (0.02 N H2SO4) used} \times 1000}{\text{ml of Sample taken}}$$

7. Determination of fluoride and nitrate

7.1. Fluoride

Fluoride ions have dual significant in water supplies. High concentration of F^-causes dental fluorosis (disfigurement of the teeth). At the same time, a concentration less than 0.8mg/L results

in 'dental caries'. Hence, it is essential to maintain the F^- concentration between 0.8 to 1.0mg/L in drinking water. In cases when fluoride concentration is less than 0.6 mg/l , supplementation (addition) of fluoride is necessary. Accurate determination of fluoride has increased the importance in public water supply system as a public health measure. Maintenance of an optimal fluoride concentration is essential is maintaining effectiveness and safety of the fluoridation procedure.

7.1.1. Determination

A. Preliminary Distillation

A fluoride can be separated from other non-volatile constituents in water by conversion to hydrofluoric or fluorosilic acid and sbubsequent distillation. The conversion is obtained by using a strong, high boiling acid distillation will separate most of the interfering ions of fluoride and thus cause separation of fluoride ions.

7.1.2. SPANDS method for determination of fluoride

Principle:

The spands colourimetric method is based on reaction between the fluoride and a zirconium dye. Fluoride react with the zirconium dye dissociating a portion of it into a colourless complex. As the amount of fluoride increases the colour produced becomes progressively lighter. Increasing fluoride concentration decreases the intensity of colour compelx due to bleaching action of fluoride and here the Beer's law is obeyed inversely.

7.1.3. Reagents:

1. Standard Fluoride solution:

   Dissolve 221 gm of anhydrous sodium fluoride in distilled water and dilute to 1 litre.

   1 ml = 100 µg of fluoride.
2. Working standard fluoride solution

   Take 100 ml from standard fluoride solution and dilute to 1 litre.

   1 ml = 10 µg of fluoride.
3. SPANDS reagent solution:

Dissolve 958 mg SPANDS, sodium 2-(parasulfophenylazo)-1,8-dihydroxy-3,6-naphtalene disulfonate, also called 4,5-dihydroxy-3-(parasulfophenlazo)-2,7-naphthalenedisulfonic acid trisodium salt, in distilled water and dilute to 500 ml.

4. Zirconyle acid reagent

   Dissolve 133 mg zirconyl chloride octahydrate, ($ZrOCl_2.8H_2O$) in about 25 ml distilled water. Add 350 ml conc. HCl and dilute to 500 ml with distilled water.

5. Reference Solution

   Add 10 ml of SPANDS solution to 100 ml distilled water. Dilute 7 ml conc HCl to 10 ml and add to the diluted SPANDS solution. The resulting solution, used for setting the instrument reference point (zero).

6. Sodium arsenite solution:

   Dissolve 5.0 gms Na AsO_2 and dilute to 1 L with distilled water.

7.1.4. Procedure:

Take standards of 0,10,20,30 and 40 ml of standard fluoride solution (1ml = 10 µg). Add 10 ml zicronyle reagent. Take sample volume of 50 ml. add 10 ml SPAND's reagent to all tubes for colour development, mix well and read absorption at 575 nm. Set the 0 with reference solution.

Observation Table

Concentration (µg)	Absorption at 575 nm
10	
20	
30	
40	
50	

Calculations

mg/l of fluorides = µg of fluorides plotted from graph/ml of sample taken

7.2. Nitrate

7.2.1. Introduction:

Determination of nitrate (NO_3^-) is difficult because of the relatively complex procedures required, the high probability that interfering constituents will be present and the limited

concentration ranges of the various techniques. Nitrate is the most highly oxidised form of nitrogen compounds commonly present in natural waters. Significant sources of nitrate are chemical fertilizers, decayed vegetable and animal matter, domestic effluents, sewage sludge disposal to land, industrial discharge, leachates from refuse dumps and atmospheric washout. Depending on the situation, these sources can contaminate streams, rivers, lakes and ground water. Unpolluted natural water contains minute amounts of nitrate. Excessive concentration in drinking water is considered hazardous for infants because of its reduction to nitrite in intestinal track causing methemoglobinaemia. In surface water, nitrate is a nutrient taken up by plants and converted into cell protein. The growth stimulation of plants, especially of algae may cause objectionable eutrophication.

UV spectrophotometer method

The method is useful for the water free from organic contaminants and is most suitable for drinking. Measurement of the ultraviolet absorption at 220nm enables rapid determination of nitrate. The nitrate calibration curve follows Beer's law upto 11mg/L N.

Acidification with 1N hydrochloric acid is designed to present interference from hydroxide or concentrations up to 1,000mg/L as CaCO. Chloride has no effect on the determination. Minimum detectable concentration is 40µg/L NO_3- N.

Principle

Nitrate is determined by measuring the absorbance at 220nm in sample containing 1mL of hydrochloric acid (1N) in 100mL sample. The concentration is calculated from graph from standard nitrate solution in range 1-11mg/L as N.

Apparatus and equipment

a. Spectrophotometer, for use at 220nm and 275nm with matched silica cells of 1cm or longer light path.

b. Filter: One of the following is required.

i) Membrane filter: 0.45µm membrane filter and appropriate filter assemble

ii) Paper: Acid-washed, ashless hard-finish filter paper sufficiently retentive for fine precipitates.

c. Nessler tubes, 50mL, short form.

Reagents and standards

a. Redistilled water: use redistilled water for the preparation of all solutions and dilutions.
b. Stock nitrate solution: dissolve 721.8mg anhydrous potassium and dilute to 1000 ml with distilled water. 1mL = 100 µg N = 443µg NO_3-.
c. Standard nitrate solution: dilute 100mL stock nitrate solution to 1000mL with distilled water. 1mL = 10µg NO_3 N = 44.3µg NO3.
d. Hydrochloric acid solution: HCl, 1N.
e. Aluminium hydroxide suspension: dissolve 125g potash alum in 1000mL distilled water. Warm to 60°C, add 55-60mL NH4OH and allow to stand for 1h. Decant the supernatant and wash the precipitate a number of times till it is free from Cl, NO_2 and NO3. Finally after setting, decant off as much clean liquid as possible, leaving only the concentrated suspension.

Calibration

Prepare nitrate calibration standards in the range 0 to 350µg N by diluting 1, 2, 4, 7…..35mL of the standard nitrate solution to 50mL. Treat the nitrate standards in the same manner as the samples.

Procedure

Read the absorbance or transmittance against redistilled water set at zero absorbance or 100% transmittance. Use a wavelength of 220 nm to obtain the nitrate reading and, if necessary, a wavelength of 275nm to obtain interference due to dissolved organic matter.

Calculation

For correction for dissolved organic matter, subtract 2 times the reading at 275nm from the reading at 220nm to obtain the absorbance due to nitrate. Convert this absorbance value into equivalent nitrate by reading the nitrate value from a standard calibration curve.

Nitrate N, mg/L = mg nitrate-N /mL of sample

NO3, mg/L = Nitrate N mg/L x 4.43

8. Residual Chlorine Determination

8.1. Introduction:

The prime purpose of disinfecting public water supplies and wastewater effluent is to prevent the spread of waterborne diseases.

Most of the methods for determination of free or combined chlorine are based on reaction with reducing agents. The Iodometric method is considered the standard method against other methods. The Iodometric procedure is appropriate for total chlorine concentrations greater than 1 mg/l.

8.2. Principle:

Chlorine will liberate free iodine from potassium iodide (KI) solutions. The liberated iodine is titrated with a standard solution of sodium thiosulfate ($Na_2S_2O_3$) with starch as the indicator. The reaction is preferably carried out at pH 3-4 for this concentrated acetic acid (glacial) is added into solution.

8.3. Reagents:

1. Acetic acid, concentrated (glacial)
2. Potassium idodide, KI, crystals
3. Standard sodium thiosulfate 0.1N:

   Dissolve 25g $Na_2S_2O_3.5H_2O$ in 1 L freshly boiled distilled water and standardize against potassium dichromate after at least 2 weeks storage. Add few ml of chloroform to minimize bacterial decomposition.

Standardization of sodium thiosulfate

Dissolve 4.904 g anhydrous potassium dichromate, $K_2Cr_2O_7$, in distilled water and dilute to 1000 mL. This will give you 0.1N potassium dichromate solution.

Take 10 mL of standard sodium thiosulfate 0.1N solution. Add 1g KI crystals and 1 mL of conc H_2SO_4. Titrate immediately with 0.1N sodium thiosulphate solution until faint yellow color is obtained which indicates the liberation of iodine. Add 1 mL starch indicator which will produce blue color. Titrate further till it becomes colorless. Find normality and dilute to make 0.01N sodium thiosulphate solution. Add few mL of chloroform as preservative.

4. Starch indicator solution:

   Take 1 g starch, prepare a paste in distilled water and add it to 100 ml boiling distilled water. Use the clear supernatant.

8.4. Procedure:

Select the sample volume that will require no more than 20 mL 0.01N $Na_2S_2O_3$. Take 100 ml or suitable amount of sample and dilute to 100 ml with distilled water. Add 5 ml of acetic acide, 1 g KI crystals. Titrate it with 0.01N sodium thiosulfate reagent until faint yellow color

is obtained. Add 1 ml of starch indicator solution at this stage and titrate further till it becomes colorless.

8.5. Calculation:

Total available residual chlorine = $\frac{A \times N \times 35450}{ml\ of\ sample}$

Where:

A = ml titration for sample

N = normality of $Na_2S_2O_3$

9. Measurement of SPM & RSPM in ambient air by High Volume Sampler

9.1. Introduction:

Suspended matter means all particulate matter which is too small in size to have appreciable falling velocity and which therefore persists in atmosphere for long periods.

Suspended particulate matters consist of smoke, dust, fumes and droplets of viscous liquid. Suspended particulate matter varies in size from well below 1µm to approximately 100 µm.

This arise from many sources such as incomplete combustion of solids, liquid or gaseous fuels and waste from metallurgical, chemical and refining operations, incineration and numerous other processes. Besides, natural sources also contribute suspended matter like salt water spray, pollens etc.

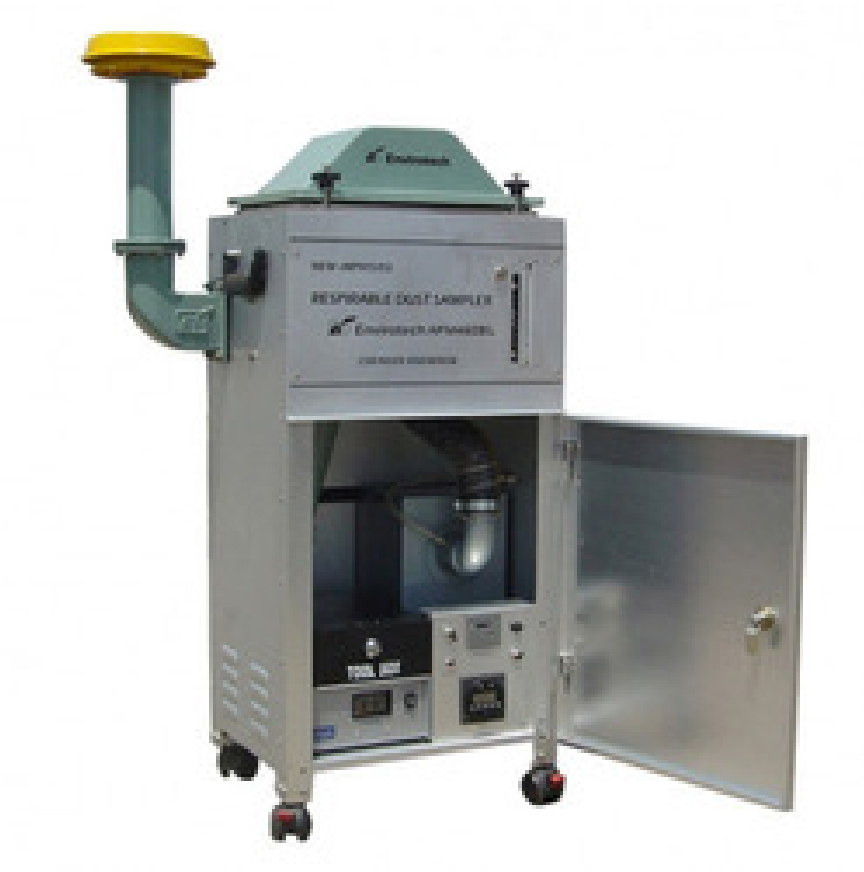

Figure 9.1: High volume sampler

Among the effects of particulate pollutants are reduction of visibility, soiling and deterioration of materials, plant damage, irritation of tissues and possible damage to health.

Table 9.1: National Ambient Air Quality Standards for Particulate matter

Sr. No.	**Pollutant**	**Time weighted average**	**Concentration in ambient air µg/m^3**		
			Industrial Area	Residential and Mix use	Sensitive Area
01	Suspended particulate matter (SPM)	Annual	80	60	15
		24 Hrs.	120	80	30

02	Repirable particulate matter	Annual	120	60	50
		24 Hrs.	150	100	75

9.2. Principle:

Air is drawn into a covered housing and through a filter by means of a high flow rate blower at a flow rate (1.13-1.7 m^3/min) that allows suspended particles having diameter less than 100µm to pass to filter surface.

Particles within size range of 100-0.1 µm diameter are collected on glass fibre filters. The mass concentration in µg/m^3 of suspended particles in ambient air is computed by measuring mass of collected particulates and volume of air sampled.

This method is applicable to measurement of mass concentration of suspended particulate matter in ambient air.

9.3. Apparatus:

1. Sampler

   It consist of 3 unit viz.

   (i)Face plate and gasket

   (ii) Filter adapter assembly and

   (iii) Motor unit

2. Sampler Shelter

   It is important that sampler shall be properly installed in a suitable shelter. The sampler is subjected to extremes of temperature, humidity and all types of air pollutants.

3. Flow measuring device

   Rotameter or u-tube monometer

4. Filter Media:

   Glass microfiber filter which is specified in official standard for environmental pollution monitoring system having high flow rate and loading capacity as well as ability to retain fine particles in range of 0.7 µm to 2.7µm.

5. Cyclone:

   Centrifugal collections employ a centrifugal force instead of gravity to separate particles from the gas stream. Cyclone consists of cylindrical shell, conical base, dust hopper and inlet where the dust laden gas enters tangentially.

9.4. Measurement and Monitoring of Airborne Particles

High volume of ambient air is sucked through a high volume sampler(HVS) and passed through a cyclone assemble where heavy particles (greater than 10µm) settle whereas less than 10µm size pass through this assembly and collected on filter paper of high volume sampler.

The difference of the weight of the filter gives the reading of PM_{10}(RSPM) and the difference of weight of the dust collected in the cyclone cup gives the reading of SPM (>10µM). Total suspended particulate matter can be worked out by adding both RSPM (<10 µm) and SPM(>10µm).

9.5. Procedure:

1. Dry filter paper at 110° C for 1-2 hours and condition the filter in the desicator and allow it to remain for 24 hours at 15-27°C and 0-50% relative humidity
2. Weigh filter paper and cyclone cup carefully on an analytical balance to four decimal places.
3. Mount filter paper in holder. Clamp it in place by fixing nuts provided for proper fixing. Tighten the nuts just to hold filter paper, do not over tight.
4. Fill the manometer with dimineralised water and set at 0-0 level.
5. Connect cyclone assembly with high volume sampler
6. Start sampler motor and record data and time. Read manometer reading and workout corresponding flow rate from calibration curve.
7. Allow the sampler to run for specified length of time of sampling period and at end of sampling period record all final readings like monometer reading, time totaliser reading and work out flow rate.
8. Remove filter from holder carefully so as not to loose any material or collected suspended particulate matter and place filter in desicator. Also remove the cyclone cup.
9. After 24 hours at 15 - 27°C, 0-58% relative humidity weighs filter and cyclone cup.
10. Difference in the weight of filter paper gives respirable suspended particulate matter and difference in weight of cyclone cup gives suspended particulate matter (10µm - 100µm) addition of two readings of respirable suspended matter gives total suspended particulate matter in ambient air.

Observations:

(i) Initial flow rate Qi =

(ii) Final flow rate Qf =

(iii) Initial weight of filter paper =

(iv) Final weight of filter paper =

(v) Initial weight of cyclone hopper cup =

(vi) Final weight of cyclone hopper cup =

Calculations:

Calculation for volume of air sample $V - \frac{Qi \times Qf}{2} \times t$

Where;

V = Total volume of air sampled, m^3

Qi = Initial air flow rate, m^3/min

Qf = Final air flow rate, m^3/min

t = sampling time, min

Calculation for RSPM (PM_{10}) $RSPM = \frac{Wf - Wi}{V} \times 10^6$

Where;

RSPM = Respirable suspended particulate matter (<10µm), µg/m^3

Wf = final weight of filter paper, g

Wi = initial weight of filter paper, g

V = total volume of air sampled, m^3

Calculation for SPM (>10 µm) $$SPM = \frac{Wf - Wi}{V} \times 10^6$$

Where;

SPM = Suspended particulate matter (>10µm), µg/m^3

Wf = final weight of cyclone hopper cup, g

Wi = initial weight of cyclone hopper cup, g

V = total volume of air sampled, m^3

10. Exhaust gas analysis for air pollutants.

Measurement of CO and HC in exhaust gas using infrared exhaust gas analyzer

10.1. Principle:

Physical phenomena may be used to determine the concentration of gaseous air pollutants. The non-dispersive infrared instrument is used to measure the gases that absorb infrared radiation for example carbonmonoxide.

The method of measurement is based on the principle of selective absorption. Instrument consists of infrared source. When the gas of interest is present, it absorbs the infrared radiation in an amount directly proportional to the molecular concentration of gas.

A particular wave length of infrared energy peculiar to a given gas will be absorbed by that gas while other wave length will be transmitted e.g. for carbon monoxide the absorption band is between 4.5μm to 5 μm so the radiation of this wavelength range will get absorbed if the CO is present and this absorption will be in proportion to the concentration of carbon monoxide present.

10.2. Measurement:

1. Connect the sampling probe to the analyser and press the measurement/standby key for measurement mode, so that clean air can be taken from the sampling probe.
2. Check that the display of CO/HC is stabilized at about the zero point, then insert the probe more than 60 cm deep into the exhaust gas pipe.
3. Insert the probe correctly to prevent entry of drain in the primary filter
4. Measured value should be read after the display has been stabilized
5. If the display is not stabilized but the zero display is stabilized, the measured value is normal.
6. Do not insert the probe unnecessarily deep into exhaust gas pipe.
7. Do not turn off the power during measurement.

10.3. Completion of measurement:

1. Don not lift up the probe when drain is collected in the primary filter, otherwise drain enters the membrane filters or sampling cell.
2. Remove the probe from the exhaust gas pipe at the completion of measurement, then check that clear air is sucked up and the display is reset to almost zero point.
3. When the analyser is left unused for a long times, press the measurement/standby key for standby mode.

Table 1: Indian Emission Standards (4-Wheel Vehicles)

Standard	Reference	YEAR	Region
India 2000	Euro 1	2000	Nationwide
Bharat Stage II	Euro 2	2001	NCR*, Mumbai, Kolkata, Chennai
		2003.04	NCR*, 13 Cities†
		2005.04	Nationwide
Bharat Stage III	Euro 3	2005.04	NCR*, 13 Cities†

		2010.04	Nationwide
Bharat Stage IV	Euro 4	2010.04	NCR*, 13 Cities†
Bharat Stage V	Euro 5	(to be skipped)	
Bharat Stage VI	Euro 6	2020.04 (proposed)[11]	Entire country
* National Capital Region (Delhi) † Mumbai, Kolkata, Chennai, Bengaluru, Hyderabad, Ahmedabad, Pune, Surat, Kanpur, Lucknow, Sholapur, Jamshedpur and Agra			

Table 2: Indian Emission Standards (2 and 3 wheelers)

Standard	Reference	Date
Bharat Stage II	Euro 2	1 April 2005
Bharat Stage III	Euro 3	1 April 2010
Bharat Stage IV	Euro 4	1 April 2012
Bharat Stage VI	Euro 6	April 2020 (proposed)

Table 3: Emission Standards for Petrol Vehicles, 4 wheelers (GVW ≤ 3,500 kg), g/km

Year	Reference	CO	HC	HC+NO_x	NO_x
1991	–	14.3–27.1	2.0–2.9	–	
1996	–	8.68–12.4	–	3.00–4.36	
1998*	–	4.34–6.20	–	1.50–2.18	
2000	Euro 1	2.72–6.90	–	0.97–1.70	
2005†	Euro 2	2.2–5.0	–	0.5–0.7	
2010†	Euro 3	2.3 4.17 5.22	0.20 0.25 0.29	–	0.15 0.18 0.21
2010‡	Euro 4	1.0 1.81 2.27	0.1 0.13 0.16	–	0.08 0.10 0.11
* for catalytic converter fitted vehicles † earlier introduction in selected regions, see Table 1 ‡ only in selected regions, see Table 1					

Table 4: Emission Standards for 3-Wheel Petrol Vehicles, g/km

Year	CO	HC	HC+NO_x
1991	12–30	8–12	–
1996	6.75	–	5.40
2000	4.00	–	2.00
2005 (BS II)	2.25	–	2.00

2010.04 (BS III)	1.25	–	1.25

Table 5: Emission Standards for 2-Wheel Petrol Vehicles, g/km			
Year	**CO**	**HC**	**HC+NO_x**
1991	12–30	8–12	–
1996	5.50	–	3.60
2000	2.00	–	2.00
2005 (BS II)	1.5	–	1.5
2010.04 (BS III)	1.0	–	1.0

11. Measurement of noise at different sources using sound meter

Use manual of the instrument to measure the noise.

Sr. No.	Noise source	Value (dB)

Observation:

12. Characterization of Municipal Solid Waste (Physical and Chemical)

12.1. Introduction:

Solid waste are all the waste arising from human and animal activities that are normally solid and that are discarded as useless or unwanted.

12.2. Types of solid waste

There are three general categories of solid waste

(i) Municipal waste

(ii) Industrial waste

(iii)Hazardous waste

(i) Municipal solid waste

Sources and types of Municipal solid waste are given in the following table

-Sources of Municipal solid waste

Source	Location of generation	Types of solid waste
Residential	Single family and multifamily dwellings, low, medium and high rise apartments etc.,	Food wastes, rubbish, ashes, special waste
Commercial	Stores, restaurants, markets, office buildings, hotels, auto repair shops, medical facilities, institutions etc.,	Food wastes, rubbish, ashes, demolition and construction wastes, special wastes, occasionally hazardous waste
Open areas	Streets, alleys, parks, vacant lots, playgrounds, highways, recreational areas etc.	Special wastes such as street sweepings, roadside litter, rubbish etc.,
Treatment plant sites	Water and wastewater and industrial treatment processes etc.,	Treatment plant wastes, principally composed of residual sludges.

(ii) Industrial solid waste

Industrial waste are those waste arising from industrial activities. It includes rubbish, ashes, demolition and construction wastes, special wastes and hazardous wastes.

(iii)Hazardous waste

Chemical, biological, flammable, explosive or radioactive wastes that pose a substantial danger, immediately or over a time to human, plant or animal life are classified as hazardous wastes. Typically this waste occurs as liquids but they are often found in form of gases, solids or sludges.

12.3. Determination of components in the field

Because of heterogeneous nature of solid wastes, determination of the composition is not an easy task. For this reasons a more generalized field procedure based on common sense and random sampling techniques has been developed as determining composition of solid waste.

The procedure involves unloading a quantity of wastes in a controlled area of a disposal site that is isolated from winds and separate from other operations. A representative residential might be a truck load resulting from a typical daily collection in a residential area.

To ensure that the result obtained are sound statically, a large enough sample must be obtained. It has been found that measurements made on a sample size of about 200 lb very insignificantly from measurement made on samples up to 1700 lb taken from the same waste load.

The following technique is followed for assessment of solid waste components.

1. Unload a truckload of wastes in a controlled area away from other operations.
2. Quarter the wasteload
3. Select one of the quarters and quarter that quarter
4. Select one the quartered quarters and separate all of the individual component of the waste into preselected components such as Food waste, paper, cardboard, plastics, rubber, textiles, leather, wood, glass metals, dirt etc.
5. Place the separated components in a container of known volume and tare mass and measure the volume and mass of each component.
6. Determine the percentage distribution of each component by mass. Typically from 100 to 200 kg of waste should be sorted to obtain a representative sample. To obtain a more representative distribution of components, samples should be collected during each season of the year.

12.4. Physical composition of Municipal solid waste

Information and data on the physical composition of solid waste are important in the selection and operation of equipment and facilities in assessing the feasibility of resource and energy recovery and in the analysis and design of disposal facilities.

The typical physical composition of Municipal solid waste is given in the following table

Component	Percent by Weight

	Range	Typical value
Food waste	6 – 26	15
Paper	25 – 45	40
Cardboard	3 – 15	4
Plastics	2 – 8	3
Textiles	0 – 4	2
Rubber	0 – 2	0.5
Leather	0 – 2	0.5
Garden trimmings	0 – 20	12
Wood	1 – 4	2
Glass	4 – 16	8
Tin Cans	2 – 8	6
Nonferrous metals	0 – 1	1
Ferrous metals	1 – 4	2
Dirt, ashes, brick etc.	0 – 10	4

12.5. Chemical composition of Municipal solid waste

Information on the chemical composition of solid waste is important in evaluating alternative processing and recovery options.

If the solid waste are to be used as fuel, the four most important properties to be known are:

1. Proximate analysis
   a. Moisture (loss at 105°C for 1h)
   b. Volatile matter (additional loss on ignition at 950°C)
   c. Ash (residue after burning)
   d. Fixed carbon (remainder)
2. Fusing point of ash
3. Ultimate analysis, percent of C(carbon), H(hydrogen), O(oxygen), N(nitrogen), S(sulfur) and ash
4. Heating value (energy value)

Typical Data on ultimate analysis of the combustible components in Municipal Solid Waste

Component	Percent by weight (dry basis)

	C	H	O	N	S	Ash.
Food Wastes	48	6.4	37.6	2.6	0.4	5.0
Paper	43.5	6	44	0.3	0.2	6
Cardboard	44	5.9	44.6	0.3	0.2	5.0
Plastic	60.0	7.2	22.8	-	-	10
Textiles	55.0	6.6	31.2	4.6	0.15	2.5
Rubber	78.0	10.0	-	2.0	-	10.0
Leather	60.0	8	11.6	10	0.4	10.0
Garden trimmings	47.8	6.0	38.0	3.4	0.3	4.5
Wood	49.5	6.0	42.7	0.2	0.1	1.5
Dirt, ashes, brick etc	26.3	3.0	2.0	0.5	0.2	68

A proximate analysis for the components of municipal solid waste as discarded is presented in table below

	Value, percent	
Component	Range	Typical
Moisture	15 – 40	20
Volatile matter	40 – 60	53
Fixed carbon	5 – 12	7
Glass,metal,ash	15 – 30	20

References:

1. Eugene W. Rice (Editor), Rodger B. Baird (Editor), Andrew D. Eaton (Editor), Lenore S. Clesceri (Editor), *Standard Methods for Examination of Water and Wastewater*, APHA
2. Guide Manual: Water and Wastewater Analysis, CPCB
3. Clair Sawyer, Perry McCarty, Gene Parkin, *Chemistry for Environmental Engineering and Science*, 5th edn, Mc Graw-Hill Higher Education

4. Manual on Municipal Solid Waste Management, CPHEEO, New Delhi.
5. IS 5182 (part 4):2004 (reaffirmed 2005) – Indian Standard, Method for measurement of air pollution, Part 4 – Suspended particulate matter
6. IS 5182 (part 14):2000 (reaffirmed 2005) – Indian Standard, Method for measurement of air pollution, Part 14 – Guidelines for planning the sampling of atmosphere
7. IS 5182 (part 23): 2006 – Indian Standard, Method for measurement of air pollution, Part 23 – Respirable suspended particulate matter (PM_{10}), cyclonic flow technique.
8. Rao, M. N. and Rao H. V. N. *Air Pollution*. Tata McGraw-Hill Publishing Co. Ltd., New Delhi 1989