CHAPTER - III
This chapter contains the methods we have adopted in carrying out this research work. Monitoring of air, water and soil quality parameters require accurate and sensitive analytical measurements.

3.1 Material and methods for air quality monitoring:

3.1.1 Analysis of SPM

Sampling site is divided into three zones as per classification prescribed by Pollution Control Board, New Delhi (ISI, 1979) and samples were collected from three sites located in different parts of the study area.

a) Site 1: Industrial area.

b) Site 2: Residential area.

c) Site 3: Commercial area.

Air was collected into a covered housing and through a pre weight Whatman GF/A filter paper for 24 hours, in a high volume air sampler by means of a high flow rate blower at a flow rate of 1.0 m$^3$/min. The mass concentration of SPM in the ambient air ($\mu$g/m$^3$) was calculated by measuring the mass collected and volume of air sampled (Naik, 2005).

The conc. of dust = \( \frac{W \times 10}{V \times T} \) $\mu$g/m$^3$

Where, $V$ = Average flow rate in m$^3$/min

$T$ = Total period of running time in minutes

$W$ = difference in final and initial weight of filter paper in gms.
High volume air sampler is a basic instrument used to monitor the ambient air quality. In these samplers, air-borne particulates are measured by passing air at high flow rate of 1.1 to 1.7 cubic meters per minute through glass fibre filter (Whatman, GF/A) paper which retains the particles. The instrument measures the volume of air sampled, while the amount of particulate collected is determined by measuring the change in weight of the filter paper as consequence of sampling. The quantity of dust collected and airflow were recorded.

3.1.2 Method for preparation of digested sample:

In case of air samples, filter paper along with the SPM was dissolved in the conc. HNO₃. After evaporating to dryness, dried extract was dissolved in 100ml distilled water and after about two hours, the solution is filtered. And thus the filtrate was ready for analysis of trace elements. After preparation of digested solution of SPM, Flame Atomic Spectrometer (Perkin Elmer Analyst200) was used to assess the metals. Standards were supplied from Perkin Elmer, USA.

3.1.3 Air quality index:

Air quality index is calculated following Tiwari and Ali method. The quality rating of each parameter is obtained by

\[ q = 100 \times \frac{V}{V_s} \]

Where \( q \) = Quality Rating

\( V \) = is the observed values of the parameter and

\( V_s \) = is the standard value recommended for the parameter.
3.2 Materials and methods for water quality monitoring:

The present study is largely confined to monitor drinking water quality around the East Guwahati industrial area of Kamrup district. Since such chemical parameters are very large and new parameters have found entry into standard methodology in recent times, monitoring whole parameters are merely an impossible task. Considering the availability of reagents and laboratory facilities, present study was undertaken with the following objectives:

3.2.1. Characterization of the drinking water quality with respect to the following physicochemical parameters -

- pH
- Conductance
- Hardness
- Chloride
- Sulphate
- Dissolved oxygen.

3.2.2. Health hazard parameters

- Nitrate
- Fluoride
- Iron
- Lead
- Manganese
- Zn.
- Bacteriological test.

3.2.3. Statistical analysis with respect to the experimental results.
3.3 Sampling Location and Collection of the samples:

In the present study, the sampling stations for drinking water were selected representing most of the area of study area. A meaningful chemical analysis largely depends on the sampling programme. The drinking water samples for physical and chemical analysis was collected in pre-cleaned 2L polyvinyl containers and brought immediately for analysis. Necessary care was taken to prevent contamination of the samples during transportation to the laboratory, storage and analysis (APHA, 1998).

3.3.1 Frequency of sampling:

For the present study, sixteen water samples were collected in the study area during the following two seasons:

- Monsoon(June, 2008 to November, 2008)
- Pre monsoon(December, 2008 to May, 2009)

3.4 Description of parameters:

3.4.1 Colour and temperature:

Colour test was carried out by comparison with a known standard. The colour of water samples of study area are observed with naked eyes by taking 50ml of the sample in a beaker. The temperature of water was collected at the time of sampling with a thermometer.
3.4.2 pH:

pH measure the hydrogen ion concentration in water. It is measured on a log scale and equal to negative log10 of hydrogen ion concentration.

\[
pH = -\log_{10}(H^+) = \log_{10}(\frac{1}{H^+})
\]

A neutral solution has a pH of 7 while pH less than 7 makes it acidic and pH greater than 7, alkaline. pH of natural water is controlled by the carbonate equilibrium involving CO₂, carbonic acid, bicarbonate and carbonate ions. For water pH is an important factor as it determines the degree of toxicity of many pollutants and the solubility of metals from bottom sediments or suspended matter (APHA, 1998). pH was measured with digital pH meter (Elico LI-127) calibrated with a solution of known pH.

3.4.3 Conductance

Conductivity is a numerical expression of the ability of an aqueous solution to carry an electric current. This ability depends on the presence of the ions, their total concentration. The physical measurement made in a laboratory determination of conductivity is usually of resistance, measured in ohms or mega ohms. The reciprocal of resistance is conductance. The resistance of a conductor is inversely proportional to its cross-sectional area and directly proportional to its length. It measures the ability to conduct a current and is expressed ohms or mhos. When the cell constant is known and applied, the measured conductance is converted to specific conductance, Kₛ, the reciprocal of the specific resistance.

\[
K_s = \frac{1}{R_s}
\]
In the international systems of units (SI), the reciprocal of ohm is the Siemens (S) and conductivity is reported as millisiemens per meter (mS/m); 1 ms/m = 10 μs/cm. Conductivity is measured using conductivity meter CM 180.

3.4.4 Hardness:

Originally, water hardness is due to the presence of dissolved salts such as magnesium and calcium carbonates, bicarbonates or sulphate. It is difficult to form lather from soap with hard water and an insoluble scum is formed. Hard water also produces in water pipes, boilers and kettles. When hardness is numerically greater than the sum of carbonate and bicarbonate alkalinity, that amount of hardness equivalent to the total alkalinity is called “carbonate hardness”; the amount of hardness in excess of this is called “noncarbonated hardness”. Hardness of water may be determined by EDTA method (Trivedy and Gowel, 1986)

Reagent:

1. EDTA solution, 0.01 m.
2. Buffer solution.
3. Erioehrome Black T
4. Sodium sulphide solution.

Procedure:

1. 50 ml of sample is taken in a conical flask.
2. 1 ml of buffer solution is added.
3. 1 ml of Na₂S solution is added.
4. After that 100 mg of Erioehrome Black T indicator is added to the solution.
5. Titrating the solution with EDTA solution, the end point is obtained.
Calculation:

Hardness, mg/L as CaCO$_3$ = ml of EDTA used $\times 1000$/ml of sample.

3.4.5 Sulphate:

Sulphate (SO$_4^{2-}$) may be pleased in water in concentration ranging from a few to several thousand milligrams per litre. Presence of excessive amount of sulphate causes disorder of elementary track leading to Diarrhea. Sulphate is generally determined by turbid metric method (Trivedy and Goel, 1986)

Reagents:

1. Conditioning reagent
2. Barium chloride

Procedure:

1. First a standard curve was formed using the standard of different concentration in spectrophotometer at the wavelength of 420nm.
2. First 100ml of sample was taken and then 5 ml of conditioning reagent was added to the sample.
3. After stirring the sample in magnetic stirrer one tablespoon of BaCl$_2$ was added.
4. Then the reading of the concentration of sample was taken in spectrophotometer at 420nm wavelength using the standard curve made earlier.
3.4.6 Nitrate:

Nitrate in large amount may cause ‘Blue babies’ to infants less than 6 months as a result of which the skin colour become dark, the infants fall sick, vomits and in extreme cases dies. The desirable limit for nitrate is 46 mg/L. Nitrate concentration can be determined by phenol Disulphonic acid method (Trivedy and Gowel, 1986).

1. Phenol Disulphonic acid
2. Silver sulphate solution.
3. Liquid ammonia.

Procedure:

1. First 50 ml of sample was taken.
2. Then silver sulphate solution was added to remove the chloride from the sample.
3. Then the sample was heated and filtered to precipitate the AgCl.
4. Then the filtrate was evaporated to dryness.
5. After cooling the residue 2ml of Phenol Disulphonic acid was added and diluted it to 50ml.
6. Then 6 ml of liquid ammonia was added to develop the yellow colour in the solution.
7. Then the reading of the concentration of sample was taken at 410nm in spectrophotometer using the standard curve.
3.4.7 Chloride:

Chloride is one of the major inorganic anions in water and wastewater. In potable water, the salty taste produced by chloride concentration is variable and dependent on the chemical composition of water. The chloride concentration is higher in wastewater than in raw water because NaCl is a common article of diet and passes unchanged through the digestive system. A high chloride concentration may harm metallic pipes and structure, as well as growing plants. Chloride can be determined by argentometric titration (Trivedy and Gowel, 1986).

Reagent:

1. Silver nitrate, 0.02 N
2. Potassium chromate, 5%.

Procedure:

1. 1.50 ml of sample is taken in a conical flask and 2 ml of K$_2$CrO$_4$ are added to it.
2. Then titrating against 0.02 N AgNO$_3$ and end point is obtained.

Calculation:

Chloride, mg/L = (ml x N) of AgNO$_3$ x 1000 x 35.5/ml of sample

Where N = normality of AgNO$_3$ used.

3.4.8 Iron

Iron in water may form rust spot on fabrics and plumbing fixtures. It may also promote the growth of certain organism causing odours and reddish brown colour to water. In some cases, it may also clog the pipeline. Permissible limit = 0.3 mg/L. Iron in drinking water can be determined by phenanthroline method using UV-visible spectrophotometer at 510nm (APHA, 1998).

Reagents:

1. Hydrochloric acid.
2. Hydroxylamine hydrochloride
3. Ammonium acetate buffer solution.
4. Phenanthroline solution.
5. Stock iron solution.

Procedure:
1. 50ml of sample was taken in a conical flask.
2. 2 ml of concentrated ammonia and 1 ml of hydroxylamine hydrochloride solution was added to the solution.
3. Then the sample was boiled to half of the contents.
4. After cooling the half sample 10ml of ammonium acetate buffer and 2ml of Phenanthroline solution was added to the solution, which gave an orange red colour to the sample.
5. Then volume of the solution was made up to 100ml and after 10 minutes the reading of the concentration of sample was taken in spectrophotometer at 510nm using the standard curve.
6. The standard curve was prepared using the known concentration in the range between 1mg/l to 4mg/l using the same procedure given above.

3.4.9 Dissolved oxygen:

Dissolved oxygen was measured by Winkler’s Iodometric method. In this method DO is allowed to react with iodide to form I₂ which is then titrated against standard Na₂S₂O₃ solution and MnSO₄ is added in alkaline medium. NaN₃ is added to avoid interferences of oxidising agents like NO₂⁻ and SO₃²⁻ (Trivedy and Gowel, 1986).
DO = (ml\times N) of sodium thiosulphate \times 8 \times 1000/V_2 \times (V_1-V)/ V_1

Where,

\( V_1 \) = volume of sample bottle
\( V_2 \) = volume of contents titrated
\( V \) = Volume of MnSO_4 and KI added.

3.4.10 Bacteriological test:

The water samples were collected in sterile glass bottle, transported on ice to the laboratory and processed within 4–6 h of collection (APHA 1998). The water quality was determined by the standard most probable number (MPN) method. Coliforms were detected by inoculation of samples into tubes of lauryl sulfate tryptose broth (LST) and incubation at 37±1°C for 48 h. The positive tubes were sub-cultured into brilliant green bile broth (BGBB) and were incubated at 44.5±1°C. Gas production in BGBB at 44.5±1°C was used for the detection of faecal coliform after 48 h incubation (Trivedy and Gowel, 1986).

3.4.11 Fluoride:

Fluoride content in the water samples were determined by using SPADNS method following the standard method of APHA, 1998. Fluoride reacts with the coloured complex of zirconyl acid and SPADNS [Sodium-2(para-sulphopherylaze) 1.8 dihydroxy-3,6 naphthalene disulphonate] to form colourless \([ZrF_6]^{2-}\) and release the dye. Fluoride can be estimated on the basis of this reaction by colorimetric measurement of the dye. Before employing the SPADNS method fluoride were separated from the water samples distilling them in presence of conc. H_2SO_4 and soft glass beads to obtain fluorosilicic acid. The absorbance measurements were carried out at 570nm using UV spectrophotometer and fluoride
concentrations were read directly operating the instrument in photometry mode calibrating against a blank and standard sodium fluoride solution.

3.4.12 Sodium and potassium:

The metals sodium and potassium were determined with a flame photometer using standard calibration procedure.

3.4.13 Trace elements (Lead, Manganese, Zinc):

All the heavy metals were determined by using Atomic Absorption spectrometer technique with the help of Perkin Elmer Analyst 200. For digestion of water samples conc. HNO₃ was used following standard method of APHA, 1998. Analysis has been done as per standard method (Direct air-acrylene flame method, p:317-318,APHA,1998 ) Chemicals used are of analytical grade. For lead (Pb) light source is electrode less discharge lamp (EDL) and for manganese and zinc, the light source is hollow cathode lamp (HCL). Wavelength and flame composition for detection of different heavy metals has been listed below:

Table 3.1: Different wavelength with flame composition.

<table>
<thead>
<tr>
<th>Instruments</th>
<th>Metals</th>
<th>Wavelength in nm</th>
<th>Flame composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Perkin Elmer Analyst 200.</td>
<td>Lead</td>
<td>217</td>
<td>Air-C₂H₂ ASS</td>
</tr>
<tr>
<td></td>
<td>Manganese</td>
<td>279.5</td>
<td>Air-C₂H₂</td>
</tr>
<tr>
<td></td>
<td>Zinc</td>
<td>213.0</td>
<td>Air-C₃H₂ ASS</td>
</tr>
</tbody>
</table>
3.4.14 Ground water quality index:

Water quality index was calculated using the following steps (G. Udayalaxmi, D. Himabindu and G. Ramadass, 2010). First step involves the calculation of quality rating/ sub index \( Q_i \) corresponding to ith parameters.

\[
Q_i = \frac{(M_i - L_i)}{(S_i - 1_i)} \times 100
\]

Second step involves the calculation of unit weight of the parameter

\[
W_i = \frac{K}{S_i}
\]

Where \( S_1, S_2, S_3, \ldots, S_i \) are the standard values of various parameters. Third step involves the calculation of overall groundwater quality index by aggregating the quality rating \( Q_i \) with the unit weight \( W_i \) linearly.

\[
GWQI = \frac{\sum Q_i W_i}{\sum W_i}
\]

3.5 Materials and methods for soil quality monitoring:

3.5.1 Soil sampling and preservation:

Soil samples were collected from the depth of about 10 cm with the help of spade by scrapping the surface layer. Nineteen soil samples from the points of specific sites were taken. Soil samples were put in thick quality polythene bag to minimise sample contamination; labelled properly for identification; sealed properly and brought to the laboratory for analysis.
3.5.2 Pre-treatment of the samples:

First soil samples were allowed to air dry in the laboratory. Then crushing and grinding of soil samples were done. The soil samples were crushed by a rubber hammer. Soil samples were sun dried for a week and then sieved through an ASTM size 50 mesh sieve to remove extra bodies present in the samples. Now the samples were ready for analysis.

3.5.3 Experimental devices used:

a) For physico-chemical analysis:

Instruments:

1. ELICO Digital pH meter model L-120.
2. Conductivity Bridge model ELICO Cm-82t.
4. UV-visible spectrometer

b) For Trace element analysis:

Instrument:


Chemicals: To digest soil samples for trace elements analysis by AAS

- Concentrated $\text{HNO}_3$. 
- Concentrated HCL
- Concentrated $\text{H}_2\text{SO}_4$
3.6 Description of the parameters:

Physical parameters:

3.6.1 Bulk density:

Bulk density is an important property which can be used to characterise the structural state of a soil. It is defined as the oven dry weight of soil per unit of its bulk volume. Bulk density of a soil indicates the degree of compactness as a measure of soil structure. Bulk density is also used to provide information on the environment, available to many soil microorganisms which live within the soil.

Measurement:

According to the procedure of M.M. Saxena, the value of bulk density of the soil is given by

\[ \text{Bulk Density, g cm}^{-3} = \frac{(W_1 - W_2)}{V} \]

Where, \( W_1 \) = wt. of the weighing bottle packed with oven dry soil.

\( W_2 \) = wt. of the empty weighing bottle.

\( V \) = Volume of the water required to fill the weighing bottle completely.

[Volume of the bottle, obtained by measuring the volume of the water required to fill it completely.]

3.6.2 Specific gravity:

Specific gravity is directly proportional to the degree of dissolved soluble material. Specific gravity of the soil particles, \( G_s \) is calculated as,

\[ G_s = \frac{W_2 - W_1}{(W_4 - W_1) - (W_3 - W_2)} \]
Where

\( W_1 = \) Wt. of vessel bottle
\( W_2 = \) Wt. of vessel and dry soil
\( W_3 = \) Wt. of vessel, soil and water.
\( W_4 = \) Wt. of vessel when full of distilled water only.

### 3.6.3 Moisture content:

Moisture content in soil maintains texture and compactness of soil and makes it habitable for plants and animals. The moisture in soil is gained from infiltration of precipitated water and irrigation. Moisture content of the soil is calculated as,

\[
\text{Moisture content (\%)} = \frac{I - F}{I} \times 100
\]

Where \( I = \) initial weight of sample (g) and \( F = \) final weight of dried sample (g).

### Chemical parameters:

#### 3.6.4 pH (Hydrogen ion concentration):

10g of air dry soil sample was taken in 100ml distilled water to make a suspension of 1:10 w/v dilution. Then pH of the suspension was determined using pH meter.

#### 3.6.5 Electrical conductivity (EC):

**Material:**

Method:

10g of air dry soil sample was taken in 100ml distilled water to make a suspension of 1:10 w/v dilution. EC of the suspension was determined using conductivity meter.

3.6.6 Alkalinity:

Reagents:

1. Sulphuric acid (0.02N): 2.8ml of Sulphuric acid was added to 1000ml of distilled water to prepare 0.1N H$_2$SO$_4$. Then 200ml of this stock solution was diluted to 1000ml to make 0.02N Sulphuric acid titrant.

2. Phenolphthalein indicator.

3. Methyl orange indicator: 0.1g of methyl orange was dissolved in 200ml distilled water.

Method:

1. 50ml of sample was taken in a flask and 2 drops of phenolphthalein was into the sample which gave pink colour to the sample.

2. Then solution was titrated against Sulphuric acid until the solution become colour less and the reading was noted.

3. Then Methyl orange indicator was added in the same flask and continued to titrate against sulphuric acid until yellow colour of the solution turn orange. Then the reading of the titrant was noted.
Calculation:

\[ P \times 100 \]

Phenolphthalein alkalinity = \[ \frac{S}{T \times 100} \]

Total alkalinity = \[ \frac{S}{T} \]

3.6.7 Organic matter:

Organic carbon present in organic matter is oxidized to CO₂ in presence of K₂Cr₂O₇ and H₂SO₄. K₂Cr₂O₇ produces nascent oxygen which combines with organic carbon producing CO₂ (Jackson M.L., 1958). Then the excess K₂Cr₂O₇ which is not reduced by organic matter is determined by titration with standard ferrous ammonium sulphate.

3.6.8 Total kjeldahl nitrogen:

The Total Kjeldahl Nitrogen method (Bremer et al., 1982.) is based on the wet oxidation of soil organic matter and botanical material using H₂SO₄ and digestion catalyst and conversion of organic nitrogen to the ammonium form. Ammonium is determined using the diffusion- conductivity technique. The method has a detection limit of approximately 0.001% N and is generally reproducible within 8%.
3.6.9 **Available phosphorus:**

The available phosphorus in soil is determined by the method of Bray and Kurtz (1945). The combination of 0.025N HCl and 0.03N NH₄F extracts acid-soluble forms of P is largely calcium phosphate and a portion of Al and Fe in acid solution and thereby release P held by Al and Fe. The P extracted from soil is a measure of labile P. P in the soil extract is determined colorimetrically using a photoelectric colorimeter after developing molybdenum blue colour using 660 μm red filter (Klett No.66), the intensity of which varies with P concentration.

3.6.10 **Potassium:**

Ammonium extract is used to extract exchangeable K in soil as exchangeable K in soil is readily displaced by NH₄⁺ ion. K in the extract is atomized into blue flame of flame photometer so that it gets excited on gaining energy and emits radiation of a certain wavelength in proportion to the concentration of K. The emitted light strikes a photocell after passing through a filter. The photocell converts light energy into electrical energy which is measured by a galvanometer. The galvanometer is first calibrated by atomizing solution varying in K concentration from 0 to 20 ppm. A direct reading flame photometer is used to determine the K concentration in soil extract.

3.6.11 **Sodium:**

Ammonium extract is used to extract exchangeable Na in soil as exchangeable Na in soil is readily displaced by NH₄⁺ ion. Na in the extract is atomized into blue flame of flame photometer so that it gets excited on gaining
energy and emits radiation of a certain wavelength in proportion to the concentration of Na. A direct reading flame photometer is used to determine the Na concentration in soil extract.

3.6.12 Calcium and magnesium:

Calcium and Magnesium in soil samples is estimated EDTA titrimetric method. EDTA is a chelating ligand which can form complex with Calcium and magnesium. In this method Murexide is used as Ca –indicator and Eriochrome Black T is used as Mg-indicator. In the presence of free Ca, Murexide gives wine red colour which turns to purple when free Ca has been completely complexed by EDTA. When Eriochrome Black T is used as indicator, EBT gives red, clear blue or green colour. As Ca EDTA complex is more stable than Mg EDTA complex, it is possible to determine Ca+Mg by EDTA titration using Mg indicator. The colour developed at the end point is the result of reaction of Mg with EBT, but it is indicative that the reaction of Ca with EDTA has been completed. The EDTA forms chelate with Ca, Mg and other metals and interferences from other metals have been removed either by chelation or by precipitation. Some metal ions like Fe$^{3+}$ are needed to be reduced prior to chelation. In Ca determination, Mg gets precipitated as Mg (OH)$_2$ on adjusting the pH of the solution to 12 or more by addition of 4N NaOH. In Mg determination, the interference of Ca is removed by precipitation of Ca by adding alcohol on introducing 1ml of 6N H$_2$SO$_4$ which effect flocculation of CaSO$_4$. 
3.6.13 Sulphate:

Sulphate in soil is determined by turbidimetric method (Bardsley and Lancaster, 1960). Sulphate in soil is extracted by 0.5N CH$_3$COONH$_4$ in acetic acid solution employing a 1:2.5 extractant (w/v) in the presence of activated charcoal free from Sulphur. The soil extract is treated with acid solution (1:1 HCl containing 20 ppm of SO$_4$- S) and crystal of BaCl$_2$ to develop BaSO$_4$ turbidity for the measurement of absorbance by UV spectrometer at 420nm. Sulphate-S is calculated as

$$\text{SO}_4^-\text{S} = \text{SO}_4\text{ in ppm} \times 0.333.$$  

3.6.14 Chloride:

The most common method for determination of chloride in soil is titrimetric method involving direct titration of the soil solution with AgNO$_3$ using K$_2$CrO$_4$ as an indicator. A 1:5 soil solution is prepared by adding 20g of soil in 100 ml distilled water and stirred mechanically for one hour at regular interval. Suspension is filtered through No.50 filter paper using Buchner funnel. 50 ml of the filtrate is titrated in an alkaline medium until the bright lemon-yellow colour just start to turn to orange. A dark reddish-orange colour will appear if excess AgNO$_3$ is added. A consistent choice of endpoint is really what is required. When Cl$^-$ in solution is exhausted through precipitation as AgCl, the red precipitate of Ag$_2$CrO$_4$ sharply signals the end point. Thiosulphate, cyanide, sulphite and sulphide are likely to interfere with this method. These can be eliminated by oxidation with 30% H$_2$O$_2$ solution before titration with AgNO$_3$. The chloride content is calculated as
% Chloride = (A×N×35.5) ml of soil solution ×2

Where, A= Volume of AgNO₃ in ml required to titrate the sample.
N= Normality of the AgNO₃.

Multiplying the values in % by 1000 give the values in mg/100g.

3.6.15 Iron:

Fe from soil is extracted by 1N CH₃COONH₄ by following the procedure of Olson (1965). The Fe in soil extract is reduced to Fe²⁺ with NH₂OH.NH₄Cl and then Fe²⁺ is reacted with 1, 10- phenanthroline to form a red coloured complex. The absorbance of the red coloured complex is measured at 510 nm wavelength using uv-visible spectrophotometer. Since 1 N CH₃COONH₄ (pH=4.8) extract in most soils are slightly coloured because of extraction of organic matter, a correction is made for the colour with each soil. This is done by running a check for each soil, to which all the reagents except orthophenanthroline is added and its absorbance is deducted from the absorbance of soil extract with all the reagents including orthophenanthroline.

3.6.16 Trace element (Pb, Mn and Zn) analysis:

3.6.16.2 Chemical: To digest soil sample for trace element analysis by AAS

- Conc. HCL
- Conc.HNO₃
- Conc. H₂SO₄

3.6.16.3 Method of digestion of samples:

1 gm of the sieved soil sample was taken in a porcelain basin and 30 ml acid mixture was added to the sample. Acid mixture was prepared as 1:2:4 proportion of the acid H₂SO₄: HCL: HNO₃ respectively. The basin containing soil sample and acid mixture was heated on a hot plate till there were no more white fumes. The content of the basin was dried. It was cooled down to normal temperature and 10 ml of hot 1:1 HCl was added to it followed by 40 ml. of distilled water. After stirring the mixture with above solution, it was filtered through Whatmann 40 filter paper. The filtrate was made upto 100 ml in a volumetric flask and taken for analysis in AAS.

3.6.16.4 Principal of Atomic absorption spectrometer (Perkin Elmer Analyst200):

A.A.S. is based on the measurement of the decreased light intensity from a source (the hollow cathode lamp) when it passes through a vapour layer of the atoms of the analysed element. The hollow cathode lamp produces intense electromagnetic radiation with a wavelength exactly the same as that absorbed by the atoms, leading to high selectivity. In atomic absorption spectroscopy, the procedure by which gaseous metal atoms are produced in the flame may be summarised as follows:
Evaporation  Vaporisation  Dissociation

M ‘X’  $\xrightarrow{\text{(gas)}}$  M ‘X’  $\xrightarrow{\text{Solution Mist Solid Gas Radiant energy \ddagger}}$  MX  $\xrightarrow{\text{M (gas) + X}}$  MX  $\xrightarrow{\text{Absorption}}$  M ‘+(gas)

3.7 Data analysis:

In the study, descriptive statistics in the form of mean, median, mode, standard deviation, coefficient of variance, confidence level, percentile, kurtosis and skewness are calculated for all parameters of soil, water and air. Coefficients of correlation are calculated to establish the relation between various parameters.

Statistical analyses of chemical parameters in the study area are summarized in tabular forms along with the graphical representation for their variation.

3.8 Health survey:

The health survey is non-experimental, descriptive research method. During our survey, different types of information have been collected from the local people regarding the health status.