Practical Manual

SOIL, WATER AND AIR POLLUTION

APS 508 3(2+1)

For

M.Sc. (Ag.) Soil Science

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Department of Soil Science

College of Agriculture

Rani Lakshmi Bai Central Agricultural University
Jhansi-284003

Practical manual

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CONTENT

EXPERIMENT–1

Objective: Sampling of sewage waters and sewage sludge

Sampling of sewage waters:

Water sampling and analysis involves the collection of water samples and measurement for chemical and biological characteristics to determine its quality.These results are compared against water quality standards in regulations and guidelines to determine its use and/or the treatment required to make the water suitable for its intended use.

These standards are defined by legislation and guidelines that govern acceptable levels of components in water that can cause health or aesthetic problems.The accuracy of water analysis is dependent on the sampling method used, the time elapsed between sampling and analysis, the techniques used in laboratory analysis and interpretation results. Water samples are used to carry out a number of different tests for water quality.

Location of Sampling

The location from which you take samples will of course depend on what you want to discover about the water.

Sampling Points:

Samples from channels are taken at two-thirds the depth of the flow at a point free back eddies. Samples of digester sludge are collected at 3 to 5 foot intervals, starting at the top and working down to avoid agitating the sludge from which the succeeding samples are taken.

Types of Sampling or Samples

There are two types of sampling techniques

1. Grab Samples
2. Composite samples

Grab Samples:

Grab sampling is just what it sounds like: all of the test material is collected at one time.
As such, grab samples reflects performance only at the point in time that the samples was collected, and then only if the samples was properly collected.

Ways to take Grab Samples:

No special equipments is needed. Usually, a sampling container is used to take the sample.The container can be dipped directly into the water or a sampling rod can be used to collect the water and fill the container.Samples are then packed in a cooler box with ice and taken for testing. Grab sampling is used to provide information about the water at one point in time.A grab sample has certain limitations. In short, a grab sample takes a snapshot of the characteristics of the water at a specific point and time, so it may not be completely representative of the entire flow. Grab Samples are most appropriate to small plans with low flows and limited staffs who cannot perform continual sampling. On the other hand, grab samples do provide an immediate sample, and are thus to be preferred for some tests. Specifically, pH, dissolved oxygen, and total residual chlorine can change very rapidly in water once the sample is removed from the flow, so grab samples are preferred for these tests.
Grab samples must be collected carefully to make them as representative as possible of the water as a whole. They should be taken at a time of day when the plant is operating near its average daily flow rate.

If grab samples are used to determine plant efficiency by collecting a raw water sample and a treated water sample, then the collection of the effluent should be delayed long enough after collection of the influent sample to allow for the raw water to pass completely through the treatment process.

Finally be aware that mixing two or more grab samples may not result in a result which averages the characteristics of the samples. Chemical reactions can take place in mixed samples which alter pH and Chlorine residual values.

COMPOSITE SAMPLES

Composite sampling involves taking number of small samples, called sub-samples, over period of time. Composite sampling consists of a collection of numerous individual discrete samples taken at regular intervals over a period of time, usually 24 hours. The material being sampled is collected in a common container over the sampling period. The analysis of this material, collected over a period of time, will therefore represent the average performance of a wastewater treatment plant during the collection period. The greatest strength of composite samples is their ability to take into account changes in flow and other characteristics of the water over time. This helps the operator gain an overall picture of the total effects that the influent will have on the treatment process and that the effluent will have on the receiving water. However, composite samples cannot be use for tests of water characteristics which change during storage (such as dissolved gases) or water characteristics which change when samples are mixed together (such as pH).

Sampling of Sewage Sludge:

1. Sampling, Preparation and Storage of Sewage Sludge. Sewage sludge is a heterogeneous, frequently unstable, mixture of dissolved and undissolved substances in water. In order to attain representative samples, the following procedures are recommended: The content of the sludge tank has to be well mixed before samples can be taken. This agitation changes certain physical characteristics by damaging the floe structure. If such physical properties are to be measured, the mixing must be done as carefully and as shortly as possible. When dealing with large sludge containers, samples should be taken at different places of the tank and then combined to provide the final sample.
2. If possible, collect the samples when the sludge is pumped, e.g. from an open duct. Equivalent volumes should be collected with equal time intervals in a defined period of time.
3. In the laboratory, the samples are drained through a 4 mm screen. When the structure of the floes is not to be considered, the sludge can also be put through a mincing machine. Immediately before analyzing, samples are stirred in accordance to the kind of information one is looking for: For parameters like water content, dried residue or density, samples are shaken thoroughly to become homogeneous. When analyzing other parameters, where the structure of the floes is of importance (e.g. sedimentation rate, filtration resistance, etc.), samples are only allowed to be stirred slowly. For better results, the sludge is carefully mixed by pouring it repeatedly from one beaker into another.
4. If the dry residue of a sample is used for additional analysis (e.g. calorific value), it has to be finely grinded (<0.2 mm)
5. Sludge samples must be kept below 4 C. They should be stored in unsealed, partly filled polyethylene bottles. For certain investigations (measurement of volatile compounds), the sludge samples have to be stored in sealed glass bottles.

EXPERIMENT –2

Objective: Sampling of solid/liquid industrial wastes

An industrial waste sampling program may serve any one of several or a combination of purposes. Because sampling procedures vary with contemplated use of the findings, it is essential that objectives be defined at the outset of the study.

Sampling equipment:

- Sample containers
- Bottle.
- Autosampler

Sampling sites:

- Mains power should to be available within 2 metres of the sampling point to eliminate the need to run auto-samplers on battery power
- A flow meter pulse output should be available for direct connection to the autosampler, enabling direct flow proportional sampling. Ideally the flow meter output connection should be located with the mains power outlet box. If direct connection to the autosampler is not available, flow meter readings will need to be taken at no greater than hourly intervals during the period of autosampler operation.
- Sample points must be precisely defined and chosen to ensure that representative samples are taken.

Sampling procedures:

Grab sampling:

When sampling from open water systems such as channels or tanks, collect the sample from the middle of the stream or body of water and at mid-depth. Avoid skimming from the surface, scraping the sides or bottom, or sampling in stagnant corners. When sampling from distribution lines, the lines should be flushed before sampling to ensure the sample collected is representative of the flowing stream and not simply material which has collected in the sampling point. Regulate the flow so that no splashing occurs.

Composite sampling using autosampler:

Autosamplers should be protected from heat, direct sunlight and sources of contamination. The sample probe should be maintained in such a position in the flow stream that representative samples are obtained (mid-depth is usually ideal). Wherever possible, avoid having the probe lying on the surface or edges of the stream. When collecting samples for charging purposes, the flow meter reading must be recorded at the commencement of sampling and the date of the reading recorded as the sample date. The preferred form of sampling, especially when collecting samples for charging, is using flow proportional composites, typically taken over a 24 hour period.

EXPERIMENT –3

Objective: Sampling of polluted soils.

It commonly refers to the analysis of a soil sample to determine nutrient content, composition and other characteristics such as the acidity or pH level

Types f Sampling

1. **Disturbed sampling** : is one in which the structure of the soil has been changed sufficiently that tests of structural properties of the soil will not be representative of in-situ conditions, and only properties of the soil grains can be accurately determined.

2. **Undisturbed Sampling**: is one where the condition of the soil in the sample is close enough to the conditions of the soil in-situ to allow tests f structural properties of the soil to be used to approximate the properties of the soil in-situ.

3. **Random Sampling**: Uniform fields can be randomly sampled throughout the entire field. To see long-term trends in soil nutrient data, these points should be geo referenced with a global positioning system (GPS) receiver and sampled in these same locations in subsequent years.

4. **Grid Sampling**

Two main types of grid sampling

1. Grid-cell soil sampling randomly collects either one or multiple subsamples throughout the cell for a composite sample.
2. Grid point soil sampling collect one or multiple subsamples around a geo referenced point within a grid or at a grid intersection.

5. **Zone Sampling:**

Zone sampling is a soil sampling technique that assumes that each field contains different soil with unique soil properties and crop characteristics and therefore should be separated into unique zones of management.

6 Topographic/Geographic Unit Sampling

Fields vary in natural features such as elevation, hilltop, slopes or depressions. Topographic/geographic unit sampling assumes these features differ in soil characteristics and therefore uses these features to establish unique zones.

2 Basic types

1- Area based sampling
2- Point based sampling

EXPERIMENT –4

Objective: Determination of Dissolved Oxygen in effluent water sample

Objectives and Scope:

Dissolved oxygen content in effluent water is a base to know the purity of water. Also it is essential in the aerobic treatment of sewage and industrial waste water. Determination of DO is a base of BOD test

Principle:

When manganous sulphate is added to the water sample containing potassium iodide, manganese hydroxide is formed. This is oxidized to basic manganic oxide by the DO present in effluent sample. When sulphuric acid is added, the basic manganic oxide liberates iodine, which is equivalent to the DO originally present in the water sample. The liberated iodine is titrated with standard sodium thiosulphate solution using starch as an indicator.

Apparatus:

Glass Stoppard bottles, flasks, beakers, measuring cylinder, burette

Reagents:

(i) Standard 0.025 N sodium thiosulphate solution: Prepare 0.1 N stock solution of sodium thiosulphate ($Na_2S_2O_3$) by dissolving 24.82 gm. of salt in 1 lit. distilled water. For preservation add 3 drops of chloroform. Take 25 ml of stock solution and dilute it to 100 ml.

(ii) Alkaline potassium iodide solution: 200g KOH + 50g KI dissolved in distilled water and make volume 500 ml.

(iii) Concentrate Sulphuric acid sp.gr. 1.84

(iv) Manganese sulphate: Dissolve 200 g $MnSO_4$ in 500 ml distilled water

(v) Starch solution: 0.5g starch dissolved in 100 ml distilled water.

Procedure:

- Fill the effluent sample in 250-300 ml glass bottle. No air bubbles should be there.
- Add 1.5 ml of manganous sulphate solution followed by 1.5 ml alkaline iodide solution keeping the tip of the pipette in each case well below the liquid surface.
- Carefully replace the stopper without inclusion of air bubbles and thoroughly mix the contents by inverting and rotating the bottle several times.
- Allow the precipitate formed to settle. When the precipitate settles leaving a clear supernatant above the manganese hydroxide flock, repeat mixing second time and allow to settle till minimum approximately 100 ml clear supernatant is to be collected.
- Remove the stopper and immediately add 2 ml of concentrated H_2SO_4 by running the acid down the neck of the bottle, restopper and mix well to ensure uniform distribution of iodine in the bottle.
- Take 200 ml solution and titrate immediately against standard sodium thiosulphate solution adding 1 ml of starch as an indicator.
- At the end point, the dark blue-black colour changes to colorless.
- Note down the reading i.e. ml of standard sodium thiosulphate solution used in titration.
- **Observations:**

1	**Titre value**	**Y ml**	**____ ml**
2	**Normality of $Na_2S_2O_3$ solution**	**N**	**0.025**
3	**Volume of sample bottle**	**V_1**	**____ ml**
4	**Volume of sample titrated**	**V_2**	**___ ml**
5	**Volume of MnSO4 and KI added**	**µ ml**	**3.0 ml**

Calculation:

If whole content have been titrated :-

$$\text{Dissolved Oxygen (mg/l)} = \frac{Y \times N \times 8 \times 1000}{V_1}$$

$$= \frac{\quad}{\quad}$$

If only part of content have been titrated :-

$$\text{Dissolved Oxygen (mg/l)} = \frac{Y \times N \times 8 \times 1000}{\frac{V_2 \times (V_1 - \mu)}{V_1}}$$

$$= \frac{\quad}{\quad}$$

EXPERIMENT –5

Objective : Determination of total dissolved solids (TDS) in effluent sample.

Total dissolved solids

Total dissolved solids (TDS) denote mainly the various kinds of mineral present in water sample. They do not contain any gaseous or colloidal fraction. They can be measured as the residue left after evaporation of the filtered sample.

Procedure

Take an evaporating dish, crucible or beaker, dry it and weigh till constant weight. Filter the water sample through whatman paper so that there is left no turbidity i.e. filtrate is as much clear as possible. Evaporate the clear filtrate in the evaporating dish or other container on a water bath. Heat at 105^0C for 1 hour in an oven. Cool in a dessicater and take the final weight. Calculate TDS as follows:

Observations:

(1)Final weight of the container (g): A _____

(2) Initial weight of the container (g): B _____

(3) Volume of the sample evaporated (ml): V _____

Calculation:

$$\text{TDS as mg/l} = \frac{(A-B) \times 1000 \times 1000}{V}$$

Where, A–final weight of the container (g),

B–Initial weight of the container (g),

V–Volume of the sample evaporated (ml).

EXPERIMENT –6

Objective: Determination of chemical oxygen demand (COD) effluents.

Scope:

COD is an indicator of quality of effluent and the base to decide the capacity/type of effluent treatment plant for an industry. The COD is a measure of oxygen equivalent to that portion of organic matter present in the waste water sample that is susceptible to oxidation by potassium dichromate. This is an important and quickly measurable parameter for stream, sewage and industrial waste samples to determine their pollution strength.

Principle:

Most of the organic matter decomposes and produces CO_2, water and NH_3 when boiled with a mixture of potassium dichromate and sulphuric acid. A sample is refluxed with a known amount of potassium dichromate in sulphuric acid medium and excess of dichromate is titrated against ferrous ammonium sulphate. The amount of dichromate consumed is proportional to the oxygen required to oxidize the organic matter.

Apparatus:

I. COD reflux unit consisting of flat bottom flask with round glass mouth andcondensers.
II. Hot water bath
III. Pipette, burette, measuring cylinder.

Reagent:

1. 0.25 N potassium dichromate solution: Dissolve 12.259 gm of AR grade $K_2C\ r_2O_7$ (Previously dried at 103 ^{0}C) in distilled water. Add about 120 gm of H_2SO_4.
2. Sulphuric acid- silver sulphate regent: Add 5.5 gm of Ag_2SO_4 to 1 kg of concentrated H_2SO_4. Keep it overnight.
3. Standard 0.1 N Ferrous ammonium sulphate solution: Dissolve 39.22 gm of ferrous ammonium sulphate in 300 ml of distilled water containing 10 ml of concentrated H_2SO_4 and dilute the solution to one liter. Make this solution freshly or titrate against the standard chromic acid each day.
4. Ferroin indicator: Dissolve 0.695 g of ferrous sulphate ($FeSO_4.7H_2O$) and 1.485 g of 1-10 Phenonthroline in distilled water to make 100 ml indicator solution.
5. Dry powder of silver sulphate.
6. Dry powder of mercuric sulphate.
7. Concentrated H_2SO_4 sp. Gr. 1.84

Procedure:

I. Take 20 ml of sample in the flask of reflux unit and add 10 ml of potassium dichromate soln., a pinch of each silver sulphate and mercuric sulphate and 30 ml of sulphuric acid.
II. Attach Liebig condenser to the mouth of flask on a hot water bath for at least two hours to reflux the contents.
III. Cool the flask, detach from the unit and dilute its contents to about 150 ml by adding distilled water.
IV. Add 2-3 drops of ferroin indicator and titrate against Ferrous Ammonium Sulphate soln. At the end point blue green colour of contents changes to reddish blue. (T).
V. Run simultaneously distilled water blank in similar manner. (B)

Observations:

1	Volume of sample taken		20.0 ml
2	Burette reading with 0.1 N Ferrous ammonium sulphate for the sample	S	_____ml
3	Burette reading with 0.1 N Ferrous ammonium sulphate for the blank	B	_____ml
4	Net 0.1 N Ferrous ammonium sulphate for 20.0 ml sample	(B-S)	_____ml

Calculation:

$$\text{COD (mg/liter)} = \frac{(B - S) \times \text{Normality of FAS} \times 1000 \times 8}{\text{Volume of sample taken (ml)}}$$

Equivalent weight of oxygen is 8.

EXPERIMENT –7

Objective: Analysis of biological demand (BOD) in effluents.

BOD is the best test for assessing the organic pollution and the base to decide the capacity/ type of effluent treatment plant for an industry. It represents the quantity of oxygen required by bacteria and other micro-organisms during the biochemical degradation and transformation of organic matter present in waste water under aerobic conditions.

Principle:

Dissolved oxygen from the effluent sample is consumed, during the oxidation of the bio-mass and reducible chemical substances, in the presence of bacteria which is measured against the original dissolved oxygen.

Apparatus:

1. Burette, pipette, measuring cylinder
2. BOD incubator
3. Glass-stoppered bottles

Reagents:

1. Standard 0.025 N sodium thiosulphate solution: Prepare 0.1 N stock solution of sodium thiosulphate by dissolving 24.82 gm. of salt in 1 lit. distilled water. For preservation add 3 drops of chloroform. Take 25 ml of stock solution and dilute it to 100 ml.
2. Concentrate Sulphuric acid sp.gr. 1.84
3. Manganese sulphate: Dissolve 200 g $MnSO_4$ in 500 ml distilled water.
4. Alkaline iodide–azide reagent: Dissolve 250 gm NaOH and 150 gm KI in distilled water. Add 5 g $NaNO_3$ in 220 ml water. Dilute whole lot to 500 ml.
5. Phosphate buffer solution: Dissolve 4.25 g potassium dihydrogen phosphate, 10.87 g of dipotassium hydrogen phosphate, 16.7 g of disodium hydrogen phosphate and 0.85 g ammonium chloride in about 250 ml dist. water and make to 500 ml (PH= 7.2).
6. Calcium chloride solution: Dissolve 13.75 g anhydrous calcium chloride in water and dilute it to make 500 ml.
7. Ferric chloride solution: Dissolve 0.125 g ferric chloride in water and dilute it to 500 ml.
8. Starch Solution: Take 0.5 g starch and prepare pest in distilled water. Make it 100 ml and boil it and allow to cool.
9. Seeding material: Supernatant liquor of domestic sewage stored for 24 to 36 hrs. at 20^oC.

Procedure:

1. Take 5 lit. of pure water.
2. Add 5 ml each of manganese sulphate, phosphate buffer, ferric chloride, and calcium chloride solution. Add 20 ml seeding material and dilute with water to 200 ml.
3. Pass to air bubble compressed for 3 to 4 hrs.
4. Neutralize the sample to 7 pH.
5. Prepare several samples by diluting 5 % to 25 %.
6. Take 6 BOD bottles. Prepare 2 for blank- for determination of initial DO (Dissolved Oxygen).
7. Add necessary diluted sample (200 ml each) in 3 bottles. Keep 2 bottles of blank & 2 having sample, in incubator at 20^0C for 5 days.
8. Take 1 blank bottle and 1 sample containing bottle. Add 1 ml manganese sulphate, 1 ml alkaline iodide azide and 1 to 2 ml cons. H_2SO_4. Stir slowly.
9. Titrate with 0.025 N sodium thiosulphate using starch as indicator. Note the burette reading.

Observations:

1	Volume of sample taken		20.0 ml
2	Dissolved oxygen of sample on 0^{th} day	X	_____
3	Dissolved oxygen of sample on 5^{th} day	Y	_____
4	Dissolved oxygen of blank on 0^{th} day	X_1	_____
5	Dissolved oxygen of blank on 5^{th} day	Y_1	_____

Calculation:

BOD (mg/liter) = $(X - Y) - (X_1 - Y_1)$ x % dilution

EXPERIMENT –8

Objective: Determination of nitrate nitrogen in effluents.

Principle: The NO_3-N is reduced to NH_3 by Devarda's alloy (Cu : Al : Zn = 50 : 45 : 5) in alkaline solution. The ammonia liberated is absorbed in known volume of 2 % boric acid solution. The amount of NH_3 absorbed is determined by titrating it with standard (0.1 N) sulphuric acid solution using mixed indicator. The end point is indicated by change of colour from sky blue to pink or wine red.

Reactions:

$Zn + 2NaOH \rightarrow Na_2ZnO_2$ (Sodium zincate) $+ 2H^+$

$Al + 2NaOH \rightarrow Na_2AlO_2$ (Sodium aluminate) $+ 2H^+$

$NO_3 + 9H^+ \rightarrow NH_3 + 3H_2O$

$3NH_3 + H_3BO_3 \rightarrow (NH_4)_3BO_3$ (Ammonium borate)

$2(NH_4)_3BO_3 + 3H_2SO_4 \rightarrow 3(NH_4)_2SO_4 + 2H_3BO_3$

(Boric acid)

Reagents:

1. Devarda's alloy : Mixture of metals in the ratio of Cu : Al : Zn :: 50 : 45 : 5 .
2. 2 % Boric acid: Dissolve 20 g H_3BO_3 in one litre volumetric flask, add about 900 ml distilled water and heat and swirl the flask until the H_3BO_3 is dissolved.
3. 2.5 % NaOH: Dissolve 25 g loose alkali (NaOH) in water and dilute to one litre volume.
4. 0.1 N H_2SO_4: Dissolve 2.8 ml concentrated H_2SO_4 in distilled water and dilutete one litre. Standardize with 0.1N Na_2CO_3 using methyl orange indicator.
5. Mixed indicator: Dissolve 0.5g Bromocresol green and 0.1 g methyl red dissolved in 100 ml of 95 % ethanol. Adjust the pH 4.5 with dilute NaOH or HCl.

Procedure:

- Pipette 25 ml of aliquot from water sample and transfer it to 1 litre of distillation flask.
- Add about 100 ml distilled water in distillation flask.
- Take 25 ml boric acid solution in a 250 ml beaker and add 4 to 5 drops of mixed indicator.
- Place the beaker containing boric acid and mixed indicator under condenser in such a way that tip of condenser should be dipped into the boric acid solution.
- Add 2 to 3 glass beads in the distillation flask.
- Add 2 gram of Devarda's Alloy in distillation flask.
- Take about 25 ml of 2.5 % NaOH solution and add into the distillation flask in such a way that it runs down from neck to the bottom without mixing. Connect the splash head immediately and circulate the water in the condenser.
- Light the burner and start heating at once to avoid the danger of sucking back.
- Continue distillation till about 150 ml of distillate is collected in the beaker. Test for NH_3 using litmus paper. Wash the tip with distilled water.
- Remove the beaker first and then put-off the burner to prevent sucking back.
- Run a blank using same procedure without addition of water sample.
- Titrate the distillate with std. 0.1 N H_2SO_4 till the pink colour is observed.
- Calculate the percent of N of the given sample of water

Observations:

3	Aliquot taken for distillation		25 ml
4	Burette reading with 0.1 N H_2SO_4 for the sample	S	_____ml
5	Burette reading with 0.1 N H_2SO_4 for the blank	B	_____ml
6	Net 0.1 required to react with NH_3 liberated from 25 ml of water sample	(S-B)	_____ml

Calculation:

1000 ml 1 N H_2SO_4 = 14 g N

1000 ml 0.1 N H_2SO_4 = 1.4 g N

1 ml 0.1 N H_2SO_4 = 0.0014 g N

$$\% N = \frac{(S - B) \times 0.0014 \times 100}{25}$$

$$\% N = (S - B) \times 0.0056$$

% N = X = _____

EXPERIMENT –9

Objective: Determination of ammonical nitrogen in effluents.

This method describes the procedure for the determination of ammonia-N in drinking, ground, and surface water; domestic and industrial waste; and biosolids (municipal sewage sludge).

Distillation of ammonia from the sample is followed by spectrophotometric analysis. Concentration range: 0.4 – 4 mg/l. If the concentration of nitrogen in ammonia is more than 4mg/L, it's necessary to dilute the sample to 200 ml with distilled water to obtain as max concentration of 4 mg/L.

Equipment:

Volumetric flasks marked at 200 ml
Volumetric flasks marked at 50 ml
Spectrophotometer UV-VIS
Cuvettes with optical path 10 mm

Reagents

- Dechlorinating reagent—Dissolve 0.35 g sodium thiosulfate (Na2S2O3*5H2O) in reagent water and dilute to 100 mL.
- Borate buffer: Dissolve 9.5 g sodium tetraborate decahydrate (Na2B4O7*10 H2O) in about 500 ml of distilled water. Add 88 mL of 0.1N NaOH solution and dilute to 1 L with reagent water.
- Boric acid 2 %: dissolve 20 g of boric acid in 1000 ml distilled water
- NaOH 6M: dissolve 24 g of NaOH in water and dilute to 100 ml
- NaOH 1 M: dissolve 40 g of NaOH in water and dilute to 1000 ml
- H_2SO_4 1N: carefully add 28 ml H_2SO_4 concentrated (d=1.84) to 500 ml of water and dilute to 1000 ml Ammonia standard solutions: dissolve 3.819 ml of ammonium chloride (NH_4Cl), dried at 110 °C, in 1 liter of distilled water (1 ml = 1 mg N-NH_3). Dilute 10 ml of this solution to 1 l with distilled water (10 mg/l N-NH_3).
- Nessler reagent for photometric reading
- Seignette salt: dissolve 50 g of sodium tartrate and potassium tetrahydrate in 30 ml of distilled water. Boil to remove ammonia residues, and after cooling dilute to 100 ml with distilled water.
- It's possible to perform this test with standard test tubes (300 ml) or with the optional 1 liter test tube (code A00001083)

Clean the distillation unit by using 200 mL of deionized water with 20 mL of borate buffer solution; distill and check if adding to 10 mL of distillate, the Nessler reagent, there is color formation. In case of color formation it's necessary to wash the distillation unit. Wash the unit 4-5 times before starting the analysis.

- Pour 200 ml of water sample into the test tube. If necessary, remove the chlorine using Dechlorinating reagent. 0.5 ml of this solution will neutralize 1 mg/L of residual chlorine in a 200 mL sample aliquot. If necessary neutralize the sample using acid (H_2SO_4 1N) or base (NaOH 1M).
- Add 10 ml of borate buffer and adjust pH to 9.5 using NaOH 6 M

Distillation and Photometric reading

Distill the samples with the following parameters (Set a customizable method):
NaOH: 0 ml

- Distillation time: 10 min
- As receiving solution, 50 ml of Boric acid (2 % p/v) has been used in a flask marked at 200 mL sample aliquot.

If necessary the sample using acid (H_2SO_4 1 N) or base (NaOH 1M).
Add 10 ml of borate buffer and adjust pH to 9.5 using NaOH 6M
Push START to begin the distillation.

Stop the analysis manually when the volume of distillate is a little bit less than 200 mL (it is important not to overcome 200 ml).
Add deionized water to dilute the distillation solution to 200 ml.
For spectrophotometric measurement, take 50 ml of distillate and add 5 drops of stabilizing solution of Seignette salt and mix well. Add 2 ml of Nessler reactive, mix well and wait for 15 min before the absorbance reading in 10 mm cuvette at 420 nm. Compare the results with a calibration curve in the range of 0.4 - 4.0 mg /l $N\text{-}NH_4$

EXPERIMENT –10

Objective: Determination of phosphorus content in effluents

Requirements- Hot plate, spectrophotometer, glass ware etc.

Reagents-

a) Perchloric acid (70%)
b) Phenolphthalein indicator- Dissolve 1.0 gm of phenolphthalein in 100 ml of ethyl alcohol and add 100 ml of distilled water.
c) Sodium hydroxide solution (1N) - Dissolve 4 gm of sodium hydroxide in distilled water to prepare 100 ml of solution.
d) Ammonium molybdate solution- Add 62ml of sulphuric acid (conc.) slowly to 80 ml of distilled water and let cool. Dissolve 5gm of ammonium molybdate in 35 ml of distilled water and mix it with sulphuric acid solution 200 ml.
e) Stannous chloride solution- Dissolves 0.5gm of stannous chloride in 2ml of conc. HCl and dilute to 20 ml distilled water.
f) Standard phosphate solution- Dissolve 4.388gm of dried anhydrous potassium hydrogen phosphate in distilled water to make the volume 1liter. Take 10 ml of this solution and add distilled water to make 1 liter of stock solution containing 1 mg P/l. Prepare standard phosphorous solution of various strengths (preferably in the range of 0.0 to 1.0 mg P/l at intervals of 0.1mg P/l) by diluting the stock solution with distilled water.

Procedure –

Take 25 ml of sample in an Erlenmeyer and evaporate to dryness. Cool and dissolve theresidue in 1 ml of perchloric acid. Heat the flask gently so that the contents become colorless. Cool and add 10 ml of distilled water and 2 drops of phenolphthalein indicator. Titrate against sodium hydroxide solution until the appearance of slight pink color. Make the volume to 25 ml by addition distilled water. Add 1 ml of ammonium molybdate solution and three drops of stannous chloride solution. A blue color will appear. Wait for 10 minutes (never more than 15 minutes) and record absorbance in spectrophotometer meter at 690 nm. Run simultaneously distilled water blank in similar manner. Process the standard phosphorous solution of different strength (reagent E) in similar manner and plot a standard curve between absorbance and concentration of standard phosphorous solutions. Deduce the total phosphorous content of sample by comparing itsabsorbance (S) with standard curve and express the result of total phosphorous in mg/l.The total particulate phosphorous can be estimated as a difference between the concentration of total phosphorous in unfiltered and filtered sample

Result- The phosphorous in given water sample was observed -------mg/l.

Precautions-

1. Glassware should be clean.
2. Prepare the standard solution carefully.

EXPERIMENT -11

Objective: Analysis of heavy metal content in effluents.

Principle

The metals are dissolved in a strong oxidizing reagent. Their concentration is determined by atomic absorption spectroscopy.

Digestion

Place about 1 g of the analytical sample to the accuracy of 1 mg into an Erlenmayer flask, and add 5 ml of digestion reagent (2 parts of cone. HNO~ p.a. and 1 part of cone. HC10. p.a.). Heat it over a laboratory sand-heat until brown nitrogen peroxide and white perchloric acid evaporates. Before the residue is evaporated to dryness, repeat the procedure until a white residue only remains in the flask. Then filter through an ash free filter into a 100 ml volumetric flask, and dilute the filtrate with 0.1 N nitric acid to 100 ml.

Calculations:

The concentration of the metal X in ppm = 100 *a/b

a = concentration of X in the solution of the digested sample [mg/1]

b = weight of the sample [g]

* Notes

1. Mercury cannot be determined with this method.
2. This digestion method is not suited for samples to be analyzed by atomic absorption using a graphite tube.
3. If dealing with very low concentrations, the digested sample has to be diluted with less than 100 ml 0.1 N nitric acid.

EXPERIMENT –12

Objective: Determination of hardness from the effluent water sample

Hardness:

This is the property of water which prevents the lather formation with soap and increases the boiling point. Calcium and magnesium are the major cations responsible for hardness. The anions which impart hardness are carbonates, bicarbonates, sulphates and chlorides.

Reagents

1. EDTA solution, 0.01 M. Dissolve 3.723 g of disodium salt of EDTA in dist. water to make volume to 1 liter. Store in polyethylene or pyrex bottle.
2. Buffer solution: Dissolve 69.5 g NH4Cl in 570 ml of NH4OH (liquid NH_3) and make one liter volume.
3. Eriochrome Black T indicator (EBT): Dissolve 0.5 g of EBT and 4.5 g of hydroxylamine hydrochloride in 100 ml of 95% ethanol.
4. Sodium sulphate solution. Dissolve 5.0 g of $Na_2S.9H_2O$ or 3.7 g of $Na_2S.5H_2O$ in 100 ml of dist. water and store in tightly closed bottles.

Procedure:

1) Take 10 ml of effluent water sample in a conical flask.
2) Add 1 ml of buffer solution. If there are higher amounts of heavy metals in sample, add 1 ml of Na_2S solution.
3) Add 2 to 3 drops of Eriochrome Black T indicator, the solution will turn to wine-red colour.
4) Titrate the contents with EDTA solution, the colour will change wine red to blue at end point.
5) Calculate the hardness of effluent water sample.

Observation and calculation:

(1) water sample taken = 10 ml

(2) EDTA used in titration =(R)_____ ml.

Hardness

(mg/l as $CaCO_3$) = $\frac{\text{(R) ml x N of EDTA x 50 x 1000}}{\text{ml water sample taken (10)}}$

EXPERIMENT –13

Objective: Determination of free carbon-dioxide from the effluent water sample

Free carbon-dioxide: This is determined by titrated the water sample against a strong alkali to pH 8.3. Following reagent are required.

Reagent:

1. **NaOH, 0.05 N:** Dissolve 40g of NaOH in boiled CO_2-free dist. water. Filter through a sintered glass filter to remove any Na_2CO_3. This is 1.0 N NaOH solution. Store it in a polythene air tight bottle. Dilute the solution 20 times to prepare 0.05 N NaOH only at the time of titration. Standardize the diluted solution with H_2SO_4, HCl or oxalic acid.
2. **Phenolphthalein indicator:** Dissolve 0.5 g of phenolphthalein in 50 ml of 95% ethanol and add 50 ml of dist. water. Add 0.05 N CO_2 – free NaOH solution drop wise, until the solution just turns faintly pink.

Procedure:

Take 10 ml of the water sample in a conical flask and add few drop of phenolphthalein indicator. The colour change to pink indicates the absence of free CO_2.In case the sample remains colorless, titrate it with 0.05 N NaOH. At the end point a pink colour appear. Calculate free CO_2 as follows.

Observation & calculation:

1. Amount of 0.05 N NaOH used in titration =(R)__ ml

2. Water sample taken = _____ ml

3. Normality of NaOH = 0.05 N

4. Equivalent weight of CO_2 = 44

Free CO_2, as mg/ l = $\frac{\text{(R) ml x N of NaOH x 1000 x 44}}{\text{ml water sample taken}}$

EXPERIMENT –14

Objective: Sampling procedure for Air.

Survey of preliminary information:

During air pollutant sampling, it is also necessary to collect information on qualitative and quantitative data on the local sources of air pollution, topography, population distribution, land use pattern, climatology, etc, depending upon the objectives of the survey or measurement campaign. For example, an area map to locate pollution sources and monitoring locations, sources of pollution situated at far distance, etc and other relevant data that describe the behavior of atmosphere for specific pollutants to be sampled may also be required.

There are two types sampling- continuous and time averaged in situ samplings, Continuous sampling is carried out by automatic sensors, optical or electrochemical and spectroscopic methods which produce continuous records. Time averaged data can also be obtained by sampling for a short time-i.e. by sampling a known volume of air for the required averaging time. Samples are then analyzed by established physical, chemical and biological methods for the concentration values which are the effective average over the period of sampling.

Sampling Locations:

Sampling locations are in general governed by factors like objectives, method of sampling and resources available. If the objective is to study health hazards and material damages, then locations should be kept close to the objects where the effects are being studied and should be kept at breathing level in the population centers, hospitals, schools, etc for vegetation, it should be at foliage level. For background concentration, sampling location should be away from the sources of pollution. It can also be done by gridding the entire area to get statistically recommended values

The number of locations however depends upon the variability of concentration over the area under survey. A spot checking may be done to decide the location besides considering practical factors.

EXPERIMENT –15

Objective: Determination of particulate matter in air sample.

Requirement- Glass jar, balance, weight box.

Method- Take 3 glass jars of known weight and put it in open area where dust fall is to be estimated. After sufficient gap of time (4 hours) collect the dust settled in each jar and weight it.
Take an average weight of dust in each jar and calculate the result. Dust fall to be expressed as weight of dust per unit area per unit time.

Particulate matter = final weight of jar – initial weight of jar

Result- The particulate matter observed was--------- µg/m3.

Precautions-

1. Beaker should be dried properly.
2. Weight the beaker paper carefully.
3. Keep the beaker at the height of approximately two meters.
4. Label the beaker properly.

EXPERIMENT –16

Objective: Determination of oxides of sulphur in air sample

Requirements- High volume sampler and spetrophotometer.

Reagents/Chemicals-

Absorbing Reagent- Potassium tetrachloromercurate (TCM 0.04M)- Dissolve 10.86 gm mercuric chloride 0.066 gm EDTA and 6.0 gm potassium chloride or sodium chloride 4.68 gm in water and bring to the mark in a 1 liter volumetric flask. The pH of this reagent should be approximately 4.0. The absorbing reagent is normally stable for six months.

Sulphamic Acid (0.6%) - Dissolve 0.6 gm sulphamic acid in 100 ml distilled water. Prepare fresh daily.

Formaldehyde (0.2%) - Dilute 5 ml formaldehyde solution (36-38%) to 1 liter with distilled water. Prepare fresh daily.

Purified Pararosaniline Stock Solution (0.2% Nominal)- Dissolve 0.500 gm of specially purified pararosaniline (PRA) in 100 ml of distilled water and keep for 2 days (48 hours).

Pararosaniline Working Solution - 10 ml of stock PRA is taken in a 250 ml volumetric flask. Add 15 ml conc. HCL and make up to volume with distilled water.

Stock Iodine Solution (0.1 N) - Place 12.7 gm iodine in a 250 ml beaker, add 40 gm potassium iodide and 25 ml water. Stir until all is dissolved then dilute to 1 litre with distilled water.

Iodine Solution (0.01 N) - Prepare approximately 0.01 N iodine solution by diluting 50 ml of stock solution to 500 ml with distilled water.

Starch Indicator Solution - Triturate 0.4 gm soluble starch and 0.002 gm mercuric iodide preservative with a little water and add the paste slowly to 200 ml boiling water. C ontinueboiling until the solution is clear, cool, and transfer to a glass-Stoppard bottle.

Stock Sodium Thiosulfate Solution (0.1 N) - Prepare a stock solution by placing 25 gm sodium thiosulphate pentahydrate in a beaker add 0.1 gm sodium carbonate and dissolve using boiled, cooled distilled water making the solution up to a final volume of 1 liter. Allow the solution to stand one day before standardizing. To standardize, accurately weigh to the nearest 0.1 mg, 1.5 gm primary standard potassium iodate dried at 180**0**C, dissolve and dilute to volume in a 500 ml volumetric flask. Into a 500 ml Iodine flask, transfer 50 ml of iodate solution by pipette. Add 2 gm potassium iodide and 10 ml of N- hydrochloric acid and stopper the flask. After 5 min, titrate with stock thiosulphate solution to a pale yellow. Add 5 ml starch indicator solution and continue the titration until the blue colour disappears. Calculate the normality of the stock solution.

Sodium Thiosulphate Titrant (0.01 N) - Dilute 100 ml of the stock thiosulphate solution to 1 litre with freshly boiled and cooled distilled water.

Sampling

Place 30 ml of absorbing solution in an impinger and sample for four hours at the flow rate of 1L/min. After sampling measure the volume of sample and transfer to a sample storage bottle.

Analysis

Replace any water lost by evaporation during sampling by adding distilled water up to the calibration mark on the absorber. Mix thoroughly, pipette out 10 ml of the collected sample into a 25 ml volumetric flask. Add 1 ml 0.6% sulphamic acid and allow reacting for 10 minutes to destroy the nitrite resulting from oxides of nitrogen. Add 2 ml of 0.2% formaldehyde solution and 2 ml pararosaniline solution and make up to 25 ml with distilled water. Prepare a blank in the same manner using 10 ml of unexposed absorbing reagent. After a 30 min colour development interval and before 60 minutes, measure and record the absorbance of samples and reagent blank at 560 nm. Use distilled water; not the reagent blank, as the optical reference.

Calibration

The actual concentration of the sulphite solution is determined by adding excess iodine and back titrating with standard sodium thiosulphate solution. To back-titrate, measure, by pipette, 50 ml of the 0.01 N iodine solutions into each of two 500 ml iodine flasks A and B. To flask a (blank) add 25 ml distilled water and into flask B (sample) measure 25 ml sulphite solution by pipette.Stopper the flasks and allow reacting for 5 minutes. Prepare the working sulphite-TCM solution at the same time iodine solution is added to the flasks. By means of a burette containing standardized 0.01N thiosulphate, titrate each flask in turn to a pale yellow. Then add 5 ml starch solution and continue the titration until the blue colour disappears.

Preparation of Standards

Measure 0.5 ml, 1.0 ml, 1.5 ml, 2.0 ml, 2.5 ml, 3.0 ml, 3.5 ml and 4.0 ml of working sulphite TCM solution in 25 ml volumetric flask. Add sufficient TCM solution to each flask to bring the volume to approximately 10 ml. Then add the remaining reagents as described in the procedure for analysis. A reagent blank with 10 ml absorbing solution is also prepared. Read the absorbance of each standard and reagent blank

Standard Curve

Plot a curve absorbance (Y axis) versus concentration (X axis). Draw a line of best fit and determine the slope. The reciprocal of slope gives the calibration factor (CF).

Calculation

Concentration of sulphite solution:

C =

+/' 2 /!- 2) 2 O

/

Where,

C = SO2 concentration in mg/ml

V1 = Volume of thiosulphate for blank (ml)

V2 = Volume of thiosulphate for sample (ml)

N = Normality of thiosulphate

K = 32000 (Milli equivalent weight SO2/µg)

V = Volume of standard sulphite solution (ml)

C (SO2 µg/m3)= (As – Ab) x CF x Vs/ Va x Vt

Where,

C SO2 = Concentration of sulphur dioxide (µg/m3)

As = Absorbance of sample

Ab = Absorbance of reagent blank

CF = Calibration factor

Va= Volume of air sampled (m3)

Vs= Volume of sample (ml)

Vt = Volume of aliquot taken for analysis (ml)

Result- SO2 in ambient air was observed--------µg/m3.

Precautions-

1. Glassware should be clean.

2. Prepare the standard solution carefully.

3. Note the reading carefully.