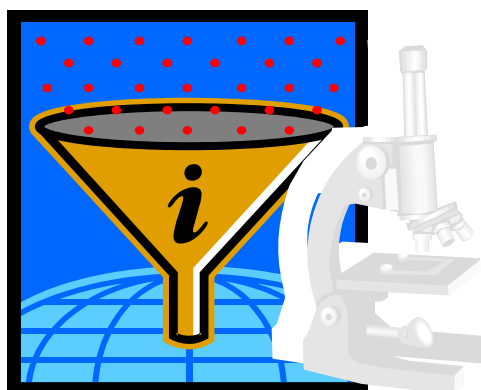
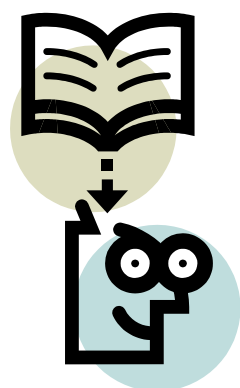


ANALYTICAL CHEMISTRY

ES 1102

LABORATORY EXERCISES



DEPARTMENT OF AGRICULTURAL ENGINEERING AND SOIL SCIENCE
FACULTY OF AGRICULTURE
RAJARATA UNIVERSITY OF SRI LANKA

General Instructions and Information

- 1) A monitor's exercise book is required and all practical must be recorded in the class.
- 2) The write up of practical should be complete and concise. It should include date, title, principle (including the equation for important reactions), method, all experimental observations, calculation and interpretation of results.
- 3) The record books should be handed over within two days after completion of the practical.
- 4) A given practical should be completed within one hour. Students are not allowed to be in the laboratory at any time other than the scheduled time.
- 5) All equipment and glassware should be kept in good condition. Glassware should be cleaned daily after use.
- 6) The students are responsible for glassware allocated to them. Any breakages should be reported immediately. The students will have to pay for any broken or lost glassware and other equipment at the end of the year.
- 7) Please be careful when using hazardous chemicals such as strong acids and bases.

Guidelines for the Preparation of Laboratory Reports

It will be a rewarding practice if you can carry out the practical, complete the calculations and enter the practical in the record book during the time provided.

Format of Laboratory Reports:

1. Date, Practical Number and Title
2. Objective/s
3. Theory / Introduction
4. Materials & Equipment
5. Procedure
6. Observations, Results and Calculations
 - Present your findings in a graphical or tabular form if possible.
 - If calculations are involved list the steps of a typical computation.
 - To obtain complete marks you have to include appropriate SI (System International) units.
 - The results should be short and concise, avoiding repetition.
7. Discussion
 - Comment on the nature of the results.
 - Comment on the nature of the experimental error.
 - List any sources of experimental error.
 - Value of application of the results.
8. Conclusion
 - Write a short conclusion of the practical with major findings.

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LABORATORY EXERCISE 01

USE AND CARE OF LABORATORY GLASSWARE

Introduction:

Different types of glassware are used in chemical analysis in laboratories. Identification, the usage and care of that glass ware are important in laboratory exercises. Most important glassware are listed below;

- | | |
|-------------------------------|-----------------------------|
| 1) Volumetric flask | 10) Burette |
| 2) Measuring cylinder | 11) Petri dish |
| 3) Conical flask | 12) Specific gravity bottle |
| 4) Funnel | 13) Glass rod |
| 5) Beaker | 14) Buchner funnel |
| 6) Watch glass | 15) Reagent bottle |
| 7) Desiccators | 16) Sampling bottle |
| 8) Test tube and boiling tube | 17) Dropping bottle |
| 9) Pipette | |

Objective:

- To familiarize with different types of laboratory glassware used for different purposes.

Exercise:

Observe and draw a rough sketch and name the glass ware given. Indicate their uses.

Cleaning Solution:

All glassware must be perfectly cleaned and free from impurities. Otherwise the results will be unreliable. Many detergents are commercially available for cleaning glassware in the laboratory. The most common method of cleaning glassware in the laboratory is filling the glassware with 'cleaning solution' and allowing to standing for several hours. A cleaning solution is a saturated solution of powdered sodium or potassium dichromate in concentrated sulphuric acid. Then the solution is poured out and the glassware is thoroughly washed with distilled water and allows draining and drying.

LABORATORY EXERCISE 02

USE AND CARE OF BASIC LABORATORY EQUIPMENT

Introduction:

Different equipment are used in a chemical laboratory for various purposes. Some of the common laboratory equipment are given below.

- 1) Burner
- 2) Tripod and wire gauze
- 3) Pair of tongs
- 4) Test tube holder
- 5) Weighing boat
- 6) Pipette filler
- 7) Mortar & pestle
- 8) Hot plate
- 9) Oven
- 10) Shaker
- 11) Stirrer
- 12) Centrifuge
- 13) Moisture can
- 14) Spatula
- 15) Test tube rack
- 16) Burette stand
- 17) Evaporating basin
- 18) Wash bottle
- 19) Water bath
- 20) Water distillation unit

Objective:

- To familiarize the students with basic laboratory equipment used in a chemical laboratory for various purposes.

Exercise:

Observe and name the different basic equipment that are found in the laboratory and indicate their specific uses.

LABORATORY EXERCISE 03

USE AND CARE OF TOP LOADING BALANCES, BURETTES AND PIPETTES

Introduction:

- **Top loading balances:** Every analysis directly or indirectly involves weighing. Therefore, correct weighing is of fundamental importance in any accurate determination. Nowadays, electronic top loading balances are used in most laboratories.

- **Burettes:** Exact measurements of volume of a solution are an important operation in quantitative analysis. Burettes are designed to deliver accurately measurable volume of a liquid. It is a glass tube that has been marked from zero to specific volume. Most commonly to 25ml or 50ml in capacity. Burettes are commonly used in titrations; so that the volume of the titrating solution needs at the end point can be measured properly.

- **Pipettes:** Pipettes are used to transfer specific known volume of a solution. Pipettes are of two kinds.

- a. Transfer pipettes: To deliver specific volume of liquid under specific conditions.
- b. Graduated or measuring pipettes: To deliver various small volumes.

Objective:

To familiarize with the operation of the electronic top loading balance.

To study the proper techniques of using burettes and pipettes in analytical chemistry.

Guidelines in using the top loading balance:

- Level the balance.
- Switch the power on and wait until zero is displayed in the digital display unit.
- Place the object to be weighed at the center of the pan.
- Take the digital reading at the end of stabilization of the balance.
- If required to take the weight of the thing only leaving the weight of the container; place the container first and press the 're-zero' or 'tare' button and wait until zero is displayed in the digital display unit.

- Then put the thing into the container and take the digital reading at the end of stabilization of the balance.

Exercise:

Weight the given objects and record the weights in the record book.

Guidelines in using a burette:

- Wash the burette and rinse with distilled water.
- Add about 10ml of the solution (to be used in the titration) using a small funnel.
- Remove the funnel and rotate the burette in a horizontal position so that the solution flows over the whole of internal surface. Discard the solution via the tip of the burette.
- Repeat this process at least twice. Never close the upper end of the burette with finger.
- Support the burette firmly on a stand vertically.
- Fill the burette with the solution to some limit while the burette is completely opened and allow it to drain off completely and repeat the process until whole air bubbles are removed.
- Then fill the burette with the solution to a little above the zero mark using a funnel.
- Remove the funnel and allow the liquid to be discharged through the stopcock until the lowest point of the liquid meniscus aligns with the zero mark.
- When delivering the liquid from burette to a conical flask use the left hand to turn the tap (if you are left handed, use the right hand). The thumb should be placed in front of the burette and the other four fingers should be behind.
- During the delivery of the liquid, shake the flask gently with the free hand.
- Bring the side of the conical flask into contact with the jet to remove any drop adhering to the jet and wash down the solution into the flask using a water jet.
- To read the position of the meniscus, the eye must be at the same level as the meniscus.
- To aid in addition the position of the meniscus a white card with a darkened (preferably black) rectangular area could be used. Place the card against the burette with the dark area just below the meniscus.
- It is advisable to move the burette with stand to stool top before taking the reading.

Exercise

Read the liquid level of the two burettes set out in the laboratory. Record the burette reading with the relevant burette number.

Guidelines in using a pipette:

- Clean and rinse the pipette with distilled water.
- Draw a portion of the liquid to a pipette using suction. Tilt and turn the pipette until the whole of internal surface has been wetted and then discard the solution. Repeat this step twice.
- Fill the pipette using suction / pipette filler to about 1 cm to 2cm above the mark.
- Then allow it to drain off the excess amount until it reaches the graduation mark by controlling the suction or pipette filler.
- Otherwise; close the upper end of the pipette with the tip of the clean dry fore finger of the dominant hand.
- Allow the liquid to run out slowly by relaxing the pressure of the finger and by carefully rotating the pipette using the other hand until the bottom of the meniscus just reaches the graduation mark.
- Hold the pipette vertically so that the mark is at the same level as the eye.
- Any drops adhering to the tip are removed by touching against a glass surface.
- Allow the liquid to run into the receiving vessel with the pipette in vertical position. The tip of the pipette should touch the wall of the vessel. When the continuous discharge has finished, hold the pipette for 15 seconds.
- Do not remove the liquid remaining in the jet of the pipette by blowing.
- Do not hold the pipette by bulb.

Exercise

Weigh an empty beaker. Pipette 10ml of distilled water into the beaker and take weight again. The weight of the water pipetted should be recorded in the record books.

LABORATORY EXERCISE 04

MAKING A STANDARD SOLUTION USING SOLID REAGENTS

Introduction:

In any analytical laboratory, it is essential to maintain stock solutions of various reagents. Some of these will be accurately known concentrations and such solutions are known as standard solutions.

In this experiment two standard solutions will be prepared from solid reagents, one from sodium carbonate (Na_2CO_3) and other one from sodium hydroxide (NaOH).

Objectives:

- To study how to prepare a standard solution using a solid reagent.
- To study how to dilute and make a standard solution from a concentrated solution of a known strength.

Procedure:

(1) Preparation of a Primary Standard Solution of Sodium Carbonate

A. Preparation of 100 ml of 0.5 M Na_2CO_3 Solution

- Weigh required amount of pure sodium carbonate in a watch glass using the top loading balance.
- Record the weight to the third decimal place.
- Transfer the contents to 100ml volumetric flask with distilled water using a small funnel.
- Use a wash bottle to remove the last traces of the substance from the watch glass and inside and outside of the funnel.
- Shake the flask until the contents are dissolved.
- Fill the water up to the mark. For the final adjustment of volume, use a dropping bottle.
- Store the solution in a glass bottle.

B. Preparation of 50 ml of 0.1M Na₂CO₃ Solution Using 0.5M Na₂CO₃ Solution

- Measure the required volume from the 0.5M Na₂CO₃ solution using a pipette.
- Transfer it to the 50ml volumetric flask.
- Fill up to the graduation mark with distilled water.

(2) Preparation of a Secondary Standard Solution of Sodium Hydroxide

A. Preparation of 100 ml of 0.5M NaOH Solution

- Weigh the required amount of pure sodium hydroxide pellets in a watch glass using the top loading balance. Record the weight to the third decimal place.
- Transfer the contents to 100ml volumetric flask with distilled water using a small funnel.
- Use a wash bottle to remove the last traces of the substance from the watch glass and inside and outside of the funnel. Shake the flask until the contents are dissolved.
- Fill the water up to the mark. For the final adjustment of volume, use a dropping bottle.
- Store the solution in a glass bottle.

B. Preparation of 50 ml of 0.1M NaOH Solution Using 0.5M NaOH Solution

- Measure the required volume from the 0.5M NaOH solution using a pipette.
- Transfer it to the 50ml volumetric flask.
- Fill up to the mark with distilled water.

Important:

- For substances that are not readily soluble in water, use a beaker to dissolve the substance initially. Heat the beaker and its contents while stirring until the solid has completely dissolved.
- Do not heat the volumetric flask under any circumstances.

Calculations:

Eg: Calculating the strength of sodium hydroxide solution

A) Strength as molarity:

$$\begin{aligned} \text{Weight of sodium hydroxide taken} &= x \text{ g} \\ \text{Moles of NaOH} &= x / 40 \text{ moles} \\ &(\text{Molecular weight of NaOH is } 40 \text{ g mol}^{-1}) \\ &\text{This amount of moles dissolved in 100ml} \\ \text{Therefore molarity of NaOH solution} &= \frac{(x / 40) \times 1000}{100} \text{ mol l}^{-1} \end{aligned}$$

B) Strength as normality:

$$\begin{aligned} \text{Equivalent weight of NaOH} &= 40 \text{ g equ}^{-1} \\ \text{Weight of NaOH taken} &= y \text{ g} \\ \text{Equivalents of NaOH} &= \frac{y}{40} \text{ equivalents} \\ \\ \text{So, Normality of NaOH solution} &= \frac{y}{40} \times \frac{1000}{100} \text{ equ l}^{-1} \end{aligned}$$

LABORATORY EXERCISE 05

MAKING A STANDARD SOLUTION USING CONCENTRATED SOLUTIONS OF KNOWN STRENGTH

Introduction:

In any analytical laboratory, some chemicals especially most acids are available in liquid form. So it is important to prepare standard solutions from those concentrated solutions of known strength.

In this experiment two standard solutions will be prepared from hydrochloric acid (HCl) and sulphuric acid (H₂SO₄).

Objectives:

- To study how to prepare a standard solution using a concentrated solution of a known strength.

Procedure:

(1) Preparation of a Standard Solution of Hydrochloric Acid

A. Preparation of 100 ml of 1M HCl Solution

- To a clean 100ml volumetric flask, add about 50ml of distilled water.
- Measure the required volume of concentrated hydrochloric acid using a pipette (Measuring cylinder is not very accurate for volume measurements) and transfer it to the 100 ml volumetric flask half filled with water.
- Fill up to the mark with distilled water.

B. Preparation of 50 ml of 0.1M HCl Solution Using 1 M HCl Solution

- Measure the required volume from the 1M HCl solution using a pipette to the volumetric flask.
- Transfer to the 50 ml volumetric flask half filled with distilled water.
- Fill up to the Mark with distilled water.

Important:

- When making standard solution of acid, always pour acid to water. Never add water to concentrated acid.

(2) Preparation of a Standard Solution of Sulphuric Acid

A. Preparation of 100 ml of 1N H₂SO₄ Solution

- To a clean 100ml volumetric flask, add about 50 ml of distilled water.
- Measure the required volume of concentrated sulphuric acid using a pipette and transfer to the volumetric flask half filled with water.
- Fill up to the mark with distilled water.

B. Preparation of 50 ml of 0.1N H₂SO₄ Solution Using 1N H₂SO₄ Solution

- Measure the required volume from the 1 N H₂SO₄ solutions using a pipette.
- Transfer to the 50 ml volumetric flask half filled with distilled water.
- Fill up to the Mark with distilled water.

LABORATORY EXERCISE 06

QUALITATIVE IDENTIFICATION OF SOME COMMON RADICALS

Introduction:

In laboratory analysis it is very important to identify the ions / common radicals from unknown chemicals. Following procedures will help to identify the ions NH_4^+ , Na^+ , K^+ , Ca^{2+} , Fe^{2+} , NO_3^- , SO_4^{2-} , Cl^- , PO_4^{3-}

Objectives:

- To study how to identify some cations and anions.

Procedure:

(I) Test for NH_4^+ ions:

- Take small amount of given sample of NH_4Cl into the boiling tube.
- Add small amount of distilled water and dissolve.
- Add few drops of 20% Sodium hydroxide (NaOH) into sample and shake it to mix.
- Heat the sample.
- Take observation using red litmus paper and lid of concentrated acid bottle during heating.

(II) Flame test for Na^+ ions:

- Take small amount of given sample of NaCl in to a watch glass.
- Take small amount of dilute Hydrochloric acid (HCl) into another watch glass.
- Expose the pencil tip to blue flame of the Bunsen burner
- Then dip the pencil tip in HCl solution
- Take sample in to a pencil tip.
- Expose the sample on pencil tip to blue flame of the Bunsen burner.
- Take observation.

(III) Flame test for K^+ ions:

- Take small amount of given sample of KCl in to a watch glass.
- Take small amount of dilute Hydrochloric acid (HCl) into another watch glass.

- Expose the pencil tip to blue flame of the Bunsen burner.
- Then dip the pencil tip in HCl solution.
- Take sample in to a pencil tip.
- Expose the sample on pencil tip to blue flame of the Bunsen burner.
- Take observation.

(IV) Flame test for Ca^{2+} ions:

- Take small amount of given sample of CaCl_2 in to a watch glass.
- Take small amount of dilute Hydrochloric acid (HCl) into another watch glass.
- Expose the pencil tip to blue flame of the Bunsen burner.
- Then dip the pencil tip in HCl solution.
- Take sample in to a pencil tip.
- Expose the sample on pencil tip to blue flame of the Bunsen burner.
- Take observation.

(V) Test for Ca^{2+} ions:

- Take small amount of given sample of CaCl into the test tube.
- Add small amount of distilled water and dissolve.
- Add small amount of ammonium oxalate solution $(\text{NH}_4)_2\text{C}_2\text{O}_4$ in to the sample.
- Take observation.

(VI) Test for Fe^{2+} ions:

- Take small amount of given sample of FeSO_4 into the test tube.
- Add small amount of distilled water and dissolve.
- Add small amount of ammonium hydroxide (NH_4OH) solution in to the sample.
- Take observation.

(VII) Brown ring test for NO_3^- ions:

- Take small amount of given sample of KNO_3 into the boiling tube.
- Add small amount of distilled water and dissolve.
- Add small amount of newly prepared ferrous sulphate (FeSO_4) solution and add few drops of concentrated sulphuric acid (H_2SO_4) along the test tube wall into the sample.
- Take observation.

(VIII) Test for SO_4^{2-} ions

- Take small amount of given sample of $(\text{NH}_4)_2\text{SO}_4$ into the test tube.
- Add small amount of distilled water and dissolve.
- Add small amount of Barium chloride (BaCl_2) in to the solution.
- Take observation.
- Add dilute Hydrochloric acid (HCl) to sample and take observation.

(IX) Test for Cl^- ions

- Take small amount of given sample of KCl into the test tube.
- Add small amount of distilled water and dissolve.
- Add small amount of Silver nitrate (AgNO_3) solution into the sample.
- Take observation.
- Then separate the sample solution in to two test tubes.
- Add dilute Nitric acid (HNO_3) in to one sample and dilute Ammonium hydroxide (NH_4OH) solution in to other sample.
- Take observation.

(X) Test for PO_4^{3-} ions

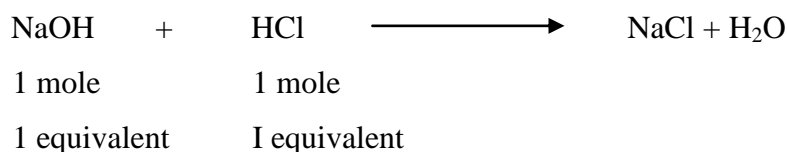
- Take small amount of given sample of NaH_2PO_4 into the boiling tube.
- Add small amount of distilled water and dissolve.
- Add concentrated Nitric acid (HNO_3) and excess ammonium molybdate solution into sample.
- Heat the contents and take observation.
- Add excess ammonia solution (NH_4OH) and again take observation.

LABORATORY EXERCISE 07

VOLUMETRIC ANALYSIS: ACID BASE TITRATION

Introduction:

Volumetric analysis refers to quantitative chemical analysis carried out by determining the volume of a standard solution (of accurately known concentration), which is required to react quantitatively with the other solution (of unknown concentration). By titrating an acid solution with a standard solution of a base, it is possible to determine the concentration of acid solution.



In this experiment the concentration of a hydrochloric acid solution will be determined by titrating with known volume of NaOH solution of known strength.

Objective:

- To study how to carry out an acid base titration and to practice the technique.

Procedure:

- Pipette 25 ml of the NaOH solution into a 250 ml conical flask.
- Add 2 drops of phenolphthalein indicator into the conical flask. Note the colour of the solution.
- Fill the burette with hydrochloric acid solution using a small funnel.
- Titrate the NaOH solution in the conical flask with hydrochloric acid solution until the end point is reached.
- Repeat the titration until the difference in reading of 3 successive titrations is less than 0.1ml.
- Calculate the concentration of hydrochloric acid solution.

Calculation:

A. Concentration as Molarity:

1. Take the average of the three-burette readings (V_2).
2. Calculate the number of moles of NaOH that was used in the titration.

The number of moles used in the titration will be

$$M_1 \cdot (25/1000) \text{ moles.}$$

$$M_1 = \text{Molarity of sodium hydroxide solution}$$

3. According to the reaction equation, 1 mole of hydrochloric acid reacts with 1 mole of sodium hydroxide. Therefore, the moles of hydrochloric acid reacted in the titration will be

$$M_1 \cdot (25/1000) \text{ moles.}$$

4. Therefore, the molarity of the hydrochloric acid (M_2) is

$$M_1 \cdot (25/1000) \cdot (1000/V_2)$$

B. Concentration as a Normality:

According to the reaction equation, 1 equivalent of HCl reacts with 1 equivalent of NaOH. Therefore, the normality of HCl solution (N_2) will be

$$N_1 V_1 = N_2 V_2$$

$$N_2 = \frac{N_1 V_1}{V_2}$$

N_1 = Normality of NaOH Solution

V_1 = Volume of NaOH Solution

N_2 = Normality of HCl Solution

V_2 = Volume of HCl Solution

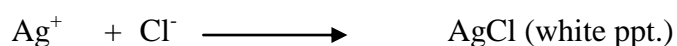
LABORATORY EXERCISE 08

VOLUMETRIC ANALYSIS: PRECIPITATION TITRATION

Introduction:

Standard silver nitrate is used for the estimation of chloride in solution. Chloride reacts with silver nitrate to form insoluble silver chloride.

The equation for the reaction is



In this titration, potassium chromate is added to the chloride solution, which act as the indicator. When all chloride has been precipitated as AgCl, the addition of more silver nitrate will cause precipitation of silver chromate which is a red precipitation.

Objective:

- To study how a precipitation reaction could be used in quantitative analysis.

Procedure:

1. Accurately weigh 0.5 – 1 g of chloride salt in a watch glass using an electronic top loading balance. Record the exact weight (to the third decimal point) in your record book.
2. Transfer it quantitatively to 250 ml volumetric flask using a funnel. Use a jet of distilled water with a wash bottle to transfer all the content to the volumetric flask.
3. Shake the flask well and make up to the mark with distilled water.
4. Take 100ml of this solution in to a clean small beaker.
5. Pipette 25ml of this solution in to a conical flask.
6. Add 1 ml of 5% potassium chromate solution using a pipette.

7. Fill the burette with silver nitrate solution using a small funnel. Adjust the volume in the burette to zero level.
8. Titrate the chloride solution in the conical flask with the silver nitrate solution. A white precipitate will be formed during the titration (precipitation of AgCl) at the end point a red precipitation of silver chromate will be formed.
9. Repeat the steps 5, 6, 7 and 8 until three successive titration agree to ± 0.1 ml.
10. Conduct a blank experiment by titration 25ml of distilled water with the same quantity of potassium chromate against the silver nitrate.
11. Calculate the concentration of chloride content in the given solution.

LABORATORY EXERCISE 09

GRAVIMETRIC ANALYSIS: DETERMINATION OF MOISTURE CONTENT OF A SOIL SAMPLE

Introduction:

Soil is a three-phase system and contains solid, water and gases. The space between the soil particles is called pore space. This pore space can act as a reservoir to store water and air. Knowing water content is important for irrigation studies. Additionally soil water influence many properties as consistency, trafficability and compatibility.

When a soil sample is obtained from the field it contains some water. Even if the soil is air-dried before sieving some moisture will remain in the sample. All your results of final analysis should be reported with respect to the oven dry weight of soil. This is done best by using a sub sample kept in the oven and obtaining the moisture content corrected for the initial moisture content in the experimental sample as shown below. When a normal laboratory oven is used, the soil is dried until there is no difference in the dry weight. This will be approximately 48 hours. When microwave ovens are available, you can use them and dry the soil for 3 minutes. When the microwave oven is used special microwave proof containers or Pyrex containers must be used. Do not use aluminum containers in the microwave oven.

Objective:

- To study how to determine the moisture content of a soil sample by gravimetric analysis.

Calculation:

$$\begin{aligned} \text{Mass of empty moisture can} &= M_1\text{g} \\ \text{Mass of moisture soil + can} &= M_2\text{g} \\ \text{Mass of oven dry moisture + can} &= M_3\text{g} \\ \text{Mass of moisture in soil} &= M_2 - M_3\text{g} \\ \text{Mass of dry soil sample} &= M_3 - M_1\text{g} \\ \text{Moisture content of sub sample} &= \frac{(M_2 - M_3)}{(M_3 - M_1)} = \theta \text{ g} \end{aligned}$$

This is called the gravimetric water content of the soil. The percentage of the gravimetric water content could be obtained by multiplying this value by 100.

Exercises:

1. Calculate the gravimetric water content for the given sample.
2. Estimate the mass of oven dry soil (soil solid) in your sample using the moisture factor as given below.

$$\text{Moisture factor} = 1 + \theta \text{ g} \quad (\text{use moisture content as a fraction for moisture factor})$$

Estimate the oven dry mass of soil sample used for the experiment by

$$\text{Oven Dry Mass} = \frac{\text{Mass of moist soil}}{\text{Moisture factor}}$$

3. Calculate the mass of fresh soil (soil of similar moisture content you used for the moisture determination) need to obtain 1 kg of oven dry soil.

LABORATORY EXERCISE 10

USE AND CARE OF ANALYTICAL INSTRUMENTS

Introduction:

Different analytical instruments are used in chemical analysis for high precision work. Identification and use of such analytical instruments is paramount important in soil and water analysis.

Objective:

- To familiarize the students with following analytical instruments.

Flame photometer

UV/ Visible spectrophotometer

Atomic Absorption Spectrophotometer

Exercise:

Observe and draw a rough sketch of aforementioned equipment. List basic applications of specified equipment.