**INDUSTRIAL ATTACHMENT REPORT**

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# DECLARATION

This attachment report is my original work prepared with no other than the indicated

Sources and support and has not been presented elsewhere for a degree or any other

Award

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# ACKNOWLEDGEMENT

I would like to acknowledge the Directorate of Geological Surveys Ministry of Petroleum and Mining, State Department of Mining administration for giving me an opportunity to be attached at the Ministry Laboratories YOU WERE ALSO ATTACHED TO ATHER SECTIONS. I also acknowledge my industrial supervisors, for helping me gain the knowledge and more so hands-on experience in handling of different equipment’s at the Ministry Laboratories.

# ABSTRACT

The Ministry of Petroleum and Mining is located along Machakos road near National Cereals and Produce Board offices in Nairobi Industrial Area. The following report is a summary of the activities partaken since the beginning of the industrial attachment from 1ST of September 2019 to 30THNovember 2019. Various analytical techniques such as Spectroscopy, Titrimetric and Gravimetric were practically done.

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**CHAPTER ONE**

# 1.0 INTRODUCTION &INDUSTRY INFORMATION

 

It was established in the year 2013. There are two departments under the ministry i.e.

1. Department of remote sensing and survey
2. Mines and geological department. The correct directorates are listed in page 3 so you can remove this

* Vision

Satisfaction of our entire customer with mineral resources and geospatial data sources

* Mission

Provide suitability of our customer with mineral resources and data service that exceeds the customer’s needs.

* Mandate

Mining and mineral development, recourses survey and remote sensing etc.

* My attachment was majorly based on the department of mines and Directorate of Geological survey where on arrival for industrial attachment I was introduced to various sections that include: Explosives &Drilling, Geophysics, Cartography, Lapidary lab, Geochemistry lab and Assay lab.

## 1.2 DIRECTORATES

1. Directorate of Mineral Management and Regulation.
2. Directorate of Geological Survey.
3. Directorate of Mineral Promotion and Value Addition.
4. Directorate of Mine Health and Safety Environment.
5. Directorate of Resource Survey and Remote Sensing.
6. Directorate Cooperate Affairs.
7. Geo Data Center and Mineral Certification Laboratory.
8. Mineral Audit Agency.
9. Policy Strategy, Research, Legal and Capacity Building.

### 1.3 MANDATE OF THE MINISTRY

1. Minerals Exploration and mining policy and Management.
2. Inventory and mapping of mineral resources.
3. Mining and minerals development
4. Policies on the management of quarrying and mining of rocks and industrial minerals
5. Management of health and safety in mines.
6. Policy around extractive industry.
7. Resource Surveys and remote sensing
8. Maintenance of geological data (research, collection, collation, analysis.

### **CHAPTER TWO**

### **2.1 DRILLLNG, EXPLOSIVES & BLASTING**

Blasting refers to the process of fracturing rock materials by use of a calculated amount of energy from explosives so that predetermined volume of material is broken.

**DRILLING.**

Is a cutting process that uses a drill bit to cut a hole of circular cross-section in solid materials? The drill bit is usually a rotary cutting tool, often multi-point. The bit is pressed against the work-piece and rotated at rates from hundreds to thousands of revolution per minute. This force the cutting edge against the work-piece, cutting off chips from the hole as it drilled.

In rock drilling the hole is usually not made through a circular cutting motion, though the bit is usually rotated. Instead, the hole is usually made by hammering a drill into the hole with quickly repeated short movements. The hammering action can be performed from outside the hole or within the hole. Drills used for horizontal drilling are called drifter drills

In rare cases, specially-shaped bits are used to cut holes of non-circular cross-section; a square cross-section is possible.

The reasons for casing are:

* To prevent the collapse.
* To prevent formation of water if you encounter.
* To avoid water loss.
* For recovery of strings.

Drilling is done:

1. To quantify.
2. To qualify.
3. To prove the presence of a certain mineral or water underground.
4. The depth of the mineral or water.

Different minerals are drilled from different angles that is either perpendicular or in angular angle. Perpendicular angle is for sedimentary rocks.

There are 4 types of bits:

1. Surface set bit it has diamond soft formation
2. Impregnated bits diamonds are embedded inside the impregnated bit. This one is for drilling.

Core boxes are used to store and preserve the drilled cores. We have two types of core boxes that are wooden core box and metallic core box. In these boxes, cores are arranged in order from the start of drilling to the end. You must indicate the depth of drilling, number of box, date and year, place of drilling and finally you have to make the head and tail of the core box.  
In the core box you have to describe the rock type, structure, shape and color of the rock. You can be able to tell the contact between the rock structures.

An explosive is any substance or device that can be made to produce a volume of rapidly expanding gas in an extremely brief period.

In a mining cycle blasting is a very essential process mostly applied in virtually all forms of mining. Black powder and dynamites were used in early ages but currently modern explosives include: ANFO (Ammonium Nitrate Fuel Oil), slurries, hybrid ANFO etc.

Blast designs on surface mining include: bench blast pattern and blast holes or initiation patterns.

The ministry of mining provides the following licenses related to explosive activities: manufacture of ANFO, special storage permit, purchase of explosives, and import/export of explosives.

## 2.2 GEOPHYSICS

**Geophysics** is the application of physics to study the Earth, oceans, atmosphere and near-Earth space. It is a broad subject that encompasses many of the major sciences – physics, astronomy, planetary science, geology, environmental science, oceanography, and meteorology.

* Magnetic method- magnetometer is used to measure the intensity of the earth’s magnetic anomalies caused by the minerals that are present in the ground
* Radiometric surveys-gamma rays spectrometers designed for detection and measurement of low level radiation from both natural occurring and manmade sources associated with radioactive elements.
* Resistivity-involves injecting current into the ground using a pair of electrodes the current causes a potential difference in the ground which is measured by a separate pair of electrodes.
* Seismic methods-uses the principle of seismology to estimate the properties of tee arts subsurface from reflected seismic waves
* Gravity surveys-can is used to detect dense bodies of rock within the host formations of the less dense wall rocks.
* Spectroscopy- uses equipment called spectrometer. The machine uses intrinsic radioactive properties of elements. Radioactive rays are used to detect elements like THz, K, and Ur etc.

## 2.3 CARTOGRAPHY

Cartography-refers to maps and the technology of map making.

Sources of information for map making.

* Existing maps
* Surveys
* Ground surveying; photographic
* Other institutions and government.

Types of maps include;

* Topographical maps-shows all geographical features within limits of scale.
* Thematic maps- show a particular thing e.g. geological maps, vegetation, and population.
* Special purpose maps-helps focus on certain details e.g. climate, topography etc.
* Bathymetric maps are used by specialists in oceans.

Map making process

Taking a photo of the area of survey then placing the photos in a computerized machine which then converts it to topographical maps

Processes involved in this department are digitization of maps which is the process of converting real information on the earth into a language a computer can understand. An application known as Arc GIS is used in the digitizing process. It interprets, analyses and and presents information on a map.

### 2.4 LAPIDARY LABORATORY

This is a laboratory that concentrates on sample crushing and mineral processing activities.

Samples are prepared here.

It is divided into four sections i.e.

1. Thin sectioning and rock dimensioning
2. Mineral processing and milling
3. X-ray fluorescence(XRF)
4. Crushing section

1. Thin sectioning and rock dimensioning

This section contains plates of different coarseness, chemicals for grinding, rock cutting machines and a petrographic microscope.

In thin sectioning carborandum is the chemical employed and is used to reduce the sample size. There are various grades of carborandum used and the petrographic microscope is used for viewing the thin section obtained. This is done to ascertain that rocks contain different minerals

1. Mineral processing and milling

* Magnetic separator

This machine was used to separate magnetic materials from the non-magnetic. It separates middling (paramagnetic) from a sample that may contain iron.

* Rapid magnetic separator

Separates magnetic materials from non-magnetic materials. It is more powerful than the magnetic separator as it picks the only magnetic materials attributed to the high fields of magnetism. Depending on the strength of magnetism each material is deposited on its specific tray.

* Tumbler machine

Used for grinding and polishing solid materials by smoothening rough surfaces.

* Rod mill / ball mill

The rod mill uses rods as grinding media with water being added to the chamber. The rods grind the ore body/sample by tumbling within the mills. After a period of time while grinding, the water solution is taken for further chemical analysis

* Concentration jig

This is an industrial machine used to separate particles based on their specific gravity. The particles of pulverized rock sample are fed over the jig bed. Due to their density, a constantly flowing air tap was opened making the materials separate from the finest to the coarse material.

* Rotan screen shaker

It is a machine used for sieve analysis. As it shakes the sample horizontally, sample particles stratify and seek critical openings in the test media

* concentration table

1. It’s mostly used to separate heavy minerals from light minerals e.g. gold, lead. Water is fed through a perforated pipe as the sample is also fed. The heavy materials move slowly to one side and the lighter materials on the other side being carried by water.

Froth floatation machine

It uses the difference of physical and chemical properties in mineral surface and relies on air bubbles buoyancy in the pulp to realize mineral separation process. This process is used to recover mineral elements in froth layers to prepare them for further analysis. This process can be used for concentration of sulfide ores. It based on preferential wetting of ore particles by oil and gangue of water. As a result the ore particles become light and rise on top in form of froth while the gangue particles become heavy and settle down.

Procedure

* The sample with the mineral of interest is added to the floatation glass and then water is added.
* The shaft is lowered so that it is deep in the floatation flask. The machine is powered on then a frothier (compounds added to stabilize the foams) like pine oil is added to help with the froth formation.
* The froth is collected and dried for further analysis.
* Brothers are added to increase froth formation. Examples include pine oil and various alcohols like Methyl isobutyl carbine (MIBC).
* Collectors increase floatability of minerals to effect separation. Examples are xanthate salts.
* Depressants prevent, temporarily or permanently, the formation of certain minerals without preventing desired mineral from being readily floated.
* Activators render floatable materials that do not correspond to collectors.

Preparation of samples for petrographic analysis.

* Small rock samples are cut using a Motta cutter, an electrically driven, manually operated machine.
* The cut rock samples are mounted on glass slides by Canadian balsam, an adhesive which makes the sample stick onto the glass slide.
* This sample is then smoothened on a smoothening slide on the dimensional table.
* Silicon carbide and Aluminum oxide is added (the mixture is called carborundum) to the smoothening plate/slide.
* The smoothening is done bit by bit, moving the rock in a circular manner to ensure uniformity, while washing in water to monitor the extent of smoothening.

The process is continued until the rock sample on the slide becomes 30 microns. The film is then taken to a microscope for further examination by geologists

.

1. X-ray fluorescence

**XRF** (**X-ray fluorescence**) is a non-destructive analytical technique used to determine the elemental composition of materials. **XRF** analyzers determine the chemistry of a sample by measuring the fluorescent (or secondary) X-ray emitted from a sample when it is excited by a primary X-ray source...

.Crushing section

Here, pulverizes and jaw crushers are used.

There are three jaw crushers i.e.

* Primary jaw crusher-crushes to 25.0 mm size
* Secondary jaw crusher-crushes to 12.5 mm size
* Laboratory jaw crusher- the sample is crashed to 6.25 mm.
* The pulverize machine is used to crush the sample to powder form i.e. approximately 100 microns. It uses circular discs that crush the sample.

## 2.5 GEOCHEMISTRY LABORATRY

Geochemistry is a lab which majorly concentrates on determination of different heavy metals which are contained in samples of different substances.

The atomic absorption spectrophotometer (AAS) is the main instrument used to detect for the presence or traces of elements in a sample and also common oxide metals

* Atomic absorption spectrophotometer (AAS)

This is a machine that quantitatively determines the presence of concentration of metals in liquid samples by using absorption of optical radiation by free atoms in the gaseous state. It was used to determine the concentration of a particular element in a sample to be analyzed. The metals that can be detected include; Fe, Cu, Al, Pb, Co, Zn, Cd etc.

There are various other small instruments such as the analytical balance for weighing and a hot plate for digestion of samples.

Major experiments in the geochemistry lab include;

Elemental analysis

Water analysis

Gypsum analysis

Full assay method

## 2.6 ASSAY LABORATORY

An **assay** is an investigative (analytic) procedure in laboratory medicine, pharmacology, environmental biology and molecular biology for qualitatively assessing or quantitatively measuring the presence, amount, or functional activity of a target entity (the analytic).

It can also be defined as examination and determination as to characteristics (such as weight, measure, or quality) or analysis (as of an ore or drug) to determine the presence, absence, or quantity of one or more components; also: a test used in this analysis.

Equipment found here includes a furnace with a capacity of heating up to 13000c, a bomb calorimeter, a carbon-Sulphur detector instrument, an oven among others.

Depending on the quality of results obtained, assays maybe classified as;

-Qualitative assay

-Semi-quantitative assay

-Quantitative assay (most performed)

The furnace is used for heating samples to high temperatures for further analysis. The bomb

**CHAPTER 3:**

3.0 SPECIFIC ACTIVITIES IN ANALYTICAL CHEMISTRY**.**

## 3.1 DETERMINATION OF Pub, Cu, Cd, Zn IN SUGAR SAMPLES

* 10.00g of a sugar sample was weighed and transferred into 100ml beaker.
* 25ml of distilled water was added and stirred to dissolve it.
* 5ml of Nitric acid was added.
* Samples were then digested on a hot plate for about 5 minutes.
* Samples were filtered into 50ml volumetric flask and topped up with distilled water up to the mark.
* The concentrations of the metals in the samples were then determined by the AAS after calibration of AAS using standards of 1ppm, 5ppm, 10ppm and a blank solution.
* Sample results in ppm(mg/kg)

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Lab No. | Sender’s Ref. | Zn | Cd | Cu | Pub |
| D314/18 | GC001 | 2.25 | ND | 1.25 | ND |
| D315/18 | GC002 | 1.60 | ND | 0.15 | 1.55 |
| D316/18 | GC003 | 0.90 | ND | 0.40 | 3.30 |
| D317/18 | GC004 | 1.30 | ND | 0.55 | 3.95 |
| D318/18 | GC005 | 1.90 | ND | 0.05 | 2.60 |

## 3.2 DETERMINATION OF COPPER CONCENTRATION IN PULVERISED ROCK SAMPLE.

* About 2.5000g of the rock sample was accurately weighed.
* 20ml of Nitric acid was added into the samples.
* Using a hot plate the sample solutions was reduced to about 10ml.
* Sample solution was filtered into a 250ml volumetric flask and topped up to the mark using distilled water.
* Samples were aspirated in the AAS to determine the concentration of copper.

= (1.35×10×250) ÷ 2.5 =1350ppm

3.3 GOLD ANALYSIS IN A ROCK SAMPLE**.**

* 20.0000g of rock samples were weighed and put in clean dry beakers.
* 20 ml of Aqua regain 1:4 was added to the samples in the beakers then they were heated on hot plate for digestion process. Samples were left to reduce to about 10ml solution.
* ?
* 30ml of distilled water was added to the cooled solutions to make them homogeneous where they were filtered in 50ml volumetric flasks using filter papers (Whitman 542 )to about 40ml solutions.
* To the solutions, 4-methyl-2-pentanone (MIBK) was added to the 50ml mark.
* Standards of gold were then prepared by preparing a 5ppm stock standard solution from the 100 ppm standard solution. 2ppm of gold standard was prepared by taking 8ml from the 5ppm stock solution and adding 20ml of MIBK. 5ppm of gold standard was prepared by taking 20ml of standard from the 5ppm stock solution and adding 20ml MIBK. 10ppm standard solution was prepared by taking 40ml of stock solution and adding 10ml of MIBK.
* The prepared standards were used to calibrate the AAS to a graph that fits the Beer-Lamberts law. The solutions were run on the AAS obeying the curve and thus the concentration of gold in the samples was obtained.

Actual concentration of gold in the rock samples was hence calculated.

3.4 FULL ASSAY ANALYSIS**.**

* 120ml plastic bottles were cleaned and dried then labeled with sample code numbers.
* 0.1000g of each pulverized rock sample was weighed and put into the bottles as per the code number indicated on then.
* 3ml of Hydrofluoric acid and 1ml of aqua regain (1:3) were added and solution left for 8hrs.
* 50ml of boric acid was then added and sample left for 30 minutes then followed by addition of 46ml of distilled water.
* 5ml of the solution sample was pipetted into a 50ml volumetric flask and topped up to the mark with 1:1 boric acid.
* AAS was calibrated with the standards of SY41/10, SY41/2 andSY4 then concentrations for SiO2, AL2O3, Fe2O3, MgO, CaO, Na20, MnO and K2O determined and recorded in result sheet.

Preparation of SY-4 standard

* 0.1000g of SY-4 powder was weighed, to its 3ml of Hydrofluoric acid and 1ml 1:3Aquaregia were added and left for 8hrs.
* 50ml Boric acid and 46ml of distilled water were added.
* From the SY-4, SY-41/2 and SY-41/10 standards were also prepared; Whose concentration for various metal oxides are as in table below:

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| STANDARD | SiO2 | Al203 | Fe2O3 | Mg | Cano | Na2O | K2O | Mn |
| SY-4 | 49.90 | 20.69 | 6.21 | 0.54 | 8.05 | 7.10 | 1.66 | 0.11 |
| SY-4½ | 24.95 | 10.35 | 3.11 | 0.27 | 4.03 | 3.55 | 0.83 | 0.06 |
| SY-41/10 | 4.99 | 2.07 | 0.62 | 0.05 | 0.81 | 0.71 | 0.17 | 0.01 |

Table 3.5 1 Preparation of SY-4 Standards

## 3.5 DETERMINATION OF CHLORIDES CONCENTRATION IN SAND SAMPLES.

* 0.5000gm of sand sample was weighed and put in a beaker.
* To the weighed sample 50ml of distilled water was added, followed by addition of 1ml Nitric acid.
* Set up was placed on hot plate and digested until it reduced to about 10ml.
* Calcium Carbonate was added until effervescence stopped thus the acidity of the resulting solution was neutralized.
* The sample was filtered in 50ml volumetric flask and topped up to the mark using distilled water.
* The Titrant 0.0141N Silver Nitrate was put into a burette.
* 25ml of the sample solution was measured and 3 drops of Potassium chromate indicator was added and solution turned yellow.
* The solution was titrated against 0.0141N Silver nitrate until blue color was observed and burette reading was recorded.

N/B: This is the procedure followed in determination of all other solid samples such as pulverized grass samples and rock samples in determination of chloride concentration.

Concentration of chlorides= (burette reading×35.450×1000×0.0141N)/volume of sample

(1.2ml×35.450×1000×0.0141N) ÷ 25ml = 23.99ppm.

**Principle behind Mohr’s method of chloride ion determination**

As silver Nitrate is slowly added; a precipitate of Gal forms;

Ag+ (as) +Cl-(as) Gal(s) white pot

The endpoint of titration occurs when all Cl- ions are precipitated. The additional Ag+ reacts with the chromate ions of the indicator K2Cr04 to form a red brown precipitate of silver chromate

2Ag+ (as) +CrO42-(aq.) Ag2CrO4(s)

The pH of sample should be between 6.5-10.If the solution is acidic gravimetric or Villard’s method should be used.

## 3.6 MOISTURE CONTENT ANALYSIS ON A ROCK SAMPLE.

* 10.00gm of a pulverized rock sample was weighed using analytical balance into a weighed and recorded pot crucible
* The sample was then put into an oven and the temperatures set at 105 0 cand left for 2 hours.
* The sample was then removed from oven, put into a desiccator and carried to the analytical balance.
* The sample was weighed and mass of crucible and sample recorded.

Moisture content= ((mass of crucible +sample before heating)-(mass after heating cuss))/initial mass\*100

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Mass of crucible | Crucible and sample before heating at 1050C | Crucible and sample after heating | Moisture content | %moisture content |
| 49.7845 | 59.2432 | 59.2432 | 0.5417 | 5.417 |

**Table 3. 1 Moisture Content Analysis on Rock Sample**

## 3.7 LOSS ON IGNITION (LOI).

* Mass of empty crucible was measured then sample of pulverized rock sample introduced on it.
* The crucible and sample was weighed and recorded then put in a desiccator
* The sample was then put in a furnace and temperatures set at 1000 0 c for 30minutes and left to cool thereafter.
* The sample was then carried in a desiccator to the analytical balance and mass of crucible and sample weighed and recorded.

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Lab No. | Mass of *empty crucible* | Mass of *crucible sample* before heating | Mass of *crucible sample* after heating | %LOI |
| D331 | 8.2080 | 9.2085 | 9.1001 | 10.83% |
| D332 | 7.0870 | 8.0931 | 8.0392 | 5.35% |
| D333 | 8.4792 | 9.4596 | 9.3936 | 6.60% |
| D334 | 7.5634 | 8.5790 | 8.5252 | 5.30% |
| D335 | 8.8433 | 9.8611 | 9.7176 | 14.10% |

**Table 3. 2 Loss on Ignition**

3.8 DETERMINATION OF SPECIFIC HEAT CAPACITY OF CHARCOAL SAMPLES**.**

* 1.0000gm of crushed charcoal sample was weighed and put in a metallic sample cup
* An ignition thread was tied at the ignition wire at the top of bomb lid and the other end buried inside the sample.
* The sample was then put into the bomb, sealed and oxygen gas filled into the bomb.
* The sample in bomb was then transferred carefully into the bucket containing 1000ml 0f water.
* The two electrodes of the colorimeter were then connected to their respective compartments and then data entered (sample ID, weight of sample and number of bombs.
* 2The start menu was then clicked an analysis began
* The final results were shown on screen with specific heat capacity of the sample which was then filled into analysis result form.

## 3.9 GRAVIMETETRIC ANALYSIS ON GYPSUM

Gypsum refers to Calcium Sulphate. (CaSO4.2H2O)

**Procedure for %sulphate analysis in a sample**

* 0.5000gm of gypsum sample was weighed using analytical balance and put in a 250ml volumetric flask.
* 1:1 HCl was added to the sample and then digested on a hot plate until it boiled.
* 100ml of boiling water was added and the sample left to boil further for 5 minutes on the hot plate.
* The sample was then left to cool and filtered on what man 542 filter paper.
* 2ml of concentrated Hall was added and sample returned on hot plate until it boiled again.
* 20ml of 12% BaCl2 was then added and sample left for 8 hours.
* The sample was then filtered over ash less what man 542 filter paper and the filter paper with precipitate dried in an oven for 30 minutes at 1050c.
* The dry filter paper was put in a weighed silicate crucible then put in a furnace to lash out at a temperature of 10000c
* The mass of the crucible and sample was then weighed and recorded.

%CaSO4.2H2O (gypsum) = ×

## 3.10 ANALYSIS OF PRECIOUS METALS IN PULVERIZED ROCK SAMPLES USING XRF ANALYSER.

The XRF analyzer was connected to a computer that had application software called Bruker.

1.5000gm of pulverized rock sample was weighed into a sample holder and placed on top of XRF analyzer and then closed.

The required settings and data such as sample lab number and application to be used (whether Geo-CChem or Alloy) were entered in the computer then the ‘Trigger pull’ menu clicked to scan the sample.

After XRF analyzer had completed the analysis; results were displayed in the screen showing the elements present and Percentage composition of each element present in the sample.

* From the Analysis one is able to conclude on the purity of a sample and whether it is authentic precious stone/metal or a synthetic product.

**How XRF works.**

* An X-ray beam with enough energy to affect the electrons in the inner shells of atoms in a sample is created by an x-ray tube inside the hand held analyzer
* The X-ray beam then interacts with the atoms in the sample by displacing electrons from inner shells of the atom. This displacement occurs as a result of interference in energy between the primary x-ray beam emitted and the binding energy that holds electrons in their proper orbits. Electrons are fixed at specific energies in their positions in an atom and this determines their orbits. Additionally the spacing between the orbital shells of an atom is unique to the atoms of each element.
* When electrons are knocked out of their orbits, they leave behind vacancies, making the atom unstable. The atom has to refill the holes/vacancies by filling them with electrons from higher orbital shells thus fluorescence.

The energy lost when electron drops from higher shell to lower shell is equivalent to energy difference between the two orbital shells and is unique to each element.

3.11 DETERMINATION OF PHOSPHATES IN WATER AND DETERGENTS**.**

* 0.2000g of the detergent sample was weighed using an analytical balance.
* To the weighed detergent 100ml of distilled water was added in 250ml beaker.
* 1ml of Sulphur acid and 5ml Nitric acid was added.
* Using a hot plate the solution was digested to about 10ml.
* 2 drops of Phenolphthalein indicator was added, followed by the addition of sodium hydroxide that controlled pH until a pink color appeared.
* 10ml of the solution was pipetted into a beaker and 10ml of Ammonium MolybdiVanadate was added and was left to form a yellow complex.
* 25ml of distilled water was added.
* Phosphorous standards of 1ppm, 5ppm and 10ppm were prepared from the 100ppm stock and from each standard 10ml were measured and placed in different beakers.
* To the 10ml of each standard measured 10ml of Ammonium MolybdiVanadate and 25ml of distilled water were added.
* Blank was also prepared by measuring 10ml of distilled water, 10 ml of Ammonium MolybdiVanadate and 25ml of distilled water were added.
* The standards and the blank were used to calibrate the UV-VIS machine. And the samples were read using the UV-VIS spectrometer in giving concentrations of phosphates present in the samples.

## 3.12 WATER ANALYSIS.

Water analyses are carried out to identify and quantify the chemical components and property of water samples. The analysis is carried out on water used in industrial processes, on waste water stream, on rivers and stream on rainfall and sea/ocean water.

**Determination of Chloride concentration in water by Titration metho**d

**Procedure**

50 ml of water sample was measured into a 250ml volumetric flask and 3 drops of indicator (K2CrO4) added to the water sample. A burette was filled with silver nitrate (AgNO3) of normality of 0.0141N and titrated against the water sample placed over a magnetic stirrer; till there was a color change of the sample and the reading noted and recorded

Formula for calculation of Cl- concentration in the sample

Concentration of chlorides = (titer value×35450×0.0141N) ÷ volume of sample taken

= 8.2×35450×0.0141N÷50

= 81.97458ppm

**Determination of heavy metals in water sample**

**Procedure**

50ml of water sample was measured and put into a beaker then 5ml of Conc. HNO3 acid added into the sample. Thereafter the contents in the beaker were heated on a hot plate until they reduced to 10ml.The sample was then filtered into a 50ml volumetric flask and topped up to the mark using warm distilled water

The concentrations of metals in the water sample were determined after calibration of AAS with the standards of 1ppm, 5ppm and 10ppm and then noting the reading for sample in the AAS.

**Determination of Total dissolved solids (TDS) and Total suspended solids (TSS)**

**Procedure**

The masses of a clean oven dried empty beaker and filter paper Whitman 542 were weighed separately and recorded.

100ml of a water sample was measured using a measuring cylinder and filtered through the filter paper into the clean dry empty beaker.

The filter paper was then dried on an oven and its mass weighed on analytical balance.

**TSS**=mass of dry filter paper after drying- mass of filter paper before filtration

= 1.2198g-1.200g = 0.0198g what about volume? Final result should be in mg/L

The beaker containing the filtrate was then heated to dryness on an oven then reweighed.

**TDS**=New mass of dried beaker- initial mass of empty beaker

= 144.7689g-144.7580g =0.109g what about volume? Final result should be in mg/L

**Determination of total water hardness**

* 50ml of water sample was measured by measuring cylinder into a 100ml beaker
* 1ml of Ammonium buffer (NH4Cl/NH4OH) solution was added.
* A Small scoop of Ferrochrome Black T indicator was added.
* Burette was filled with 0.0001M EDTA solution.
* Titration was slowly done with continuous stirring with a standard EDTA titrant, until a reddish tint disappeared.
* End point was reached when a blue color was observed and titer value was recorded.

**Determination of alkalinity in water sample**

* The pH meter was calibrated using standards of pH 4.00 and 7.00
* 50ml of each water sample was measured using a measuring cylinder into dry clean beakers
* The pH of the water samples in beakers was determined using pH meter
* 0.02N H2SO4 was titrated against the samples until the pH value was 4.5
* The titer value was recorded and used in calculation to determine alkalinity of the water samples.

Total alkalinity= (8.2\*1000)/50ml

=164mg CaCO3/L

**Determination of nitrates in water samples**

* 100ml of each water sample were measured.
* 1ml of Hydrochloric acid was added.
* Standards of nitrates of 1ppm,2ppm and 3ppm were prepared and to each 1ml of Hydrochloric acid was added and then used to calibrate the UV-VIS spectrometer and then the nitrate concentrations were thereafter read.

Nitrates concentrations= (UV-VIS reading× dilution factor × stock volume)/volume of sample

(0.88×1×1000)/100

= 8.8ppm

**Determination of Concentration of Sulphates in Water Samples**

**Preparation of Sulphate Buffer**

* 30g of Magnesium Chloride, 5g of Sodium Acetate and 1g of Potassium Nitrate were weighed using a weighing balance and placed in 1000ml volumetric flask .To it 20ml of acetic acid was added.500ml of distilled water was added as shaking was done and then topped up to the 1000ml mark with distilled water

K2SO4= (39×2) + 32 + (16×4) =174

SO4= (32+64) = 96

SO4 in K2SO4=96/174=0.55172g

1g of K2SO4=0.55172g

? = 1g of SO42-

(1×1)÷0.55172 =1.8125g

* 1.8125g of K2SO4 was weighed and placed into 1000ml volumetric flask and topped to the mark using distilled water thus, making 1000ppm of the Sulphate stock standard.

**Procedure**

* 100ml of water sample was measured and placed in 250ml beaker.20ml of Sulphate buffer was added and stirring was done. A spoonful of Barium chloride was added and stirring for 2minutes was done.
* From 1000ppm Sulphate standards, 1ppm, 5ppm, and 10ppm were prepared and to every standard spoonful of Barium chloride was added.
* The blank and standards were used to calibrate the UV-VIS spectrometer and Sulphate concentrations were then read.

## 3.13 LAPIDARY LABORATORY

In this laboratory sample preparation is carried out. Processes and machines used to achieve this include:

***Crushing process***-machines are used in breaking rocks into smaller samples that can be pulverized. Machines used in crushing are classified into 3;

* Primary jaw- crusher-breaks/crushes big rocks into smaller pieces of 25mm in size
* Secondary jaw-crusher-reduces the size of samples from primary geo crusher by half(12.5mm)
* Laboratory jaw-crusher; further reduces size of samples from secondary geo crusher by half(6.25mm thick)
* Pulverize-is a machine that further breaks samples from laboratory geo crusher into fine powder for laboratory analysis. Pulverization increases surface area for digestion and also makes it possible for the samples to dissolve in water and or other solvents.

**Concentration of samples/ore;** this involves Removal of unwanted materials (e.g. sand, clay etc.) from ore. This is being achieved by the use of separating table that separates samples from the pulverize basing on their differences in weight.

**Froth floatation;** this process is used for concentration of supplied ores. It is based on preferential wetting of ore particles by oil and gangue of water .As a result the ore particles become light and rise to the top in form of froth while the gangue particles become heavy and settle down. Thus adsorption is involved in this process.

**Electromagnetic separation;** this method of concentration is employed when either the ore or the impurities associated with it are magnetic in nature

NB. Cleaning of materials in the laboratory can be done by use of either water or high pressure cleaner or gypsum that is used to remove color in pulverization plates after pulverizing colored samples and or rocks.

**CHAPTER FOUR**

# 4.0 CHALLENGES

* Inadequate fieldwork practices.
* Lack of enough protective equipment especially in the lapidary and geochemical labs
* Lack of enough computers in the cartography lab leading to delays in practicing of digitizing and other process in the lab
* Only one AAS machine is available and students are many.
* Difficulties in having to overcome traffic jam daily in order to arrive in time at the working station.

## 4.1 RECOMMENDATIONS

* Fieldwork issue should be discussed seriously
* Purchase of adequate protective garments e.g. gas muff, gloves, clear goggles etc.
* Purchase of more computers for cartography and other labs
* More AAS machines should be put in place

## 4.2 CONCLUSION

In conclusion the attachment was a great source of experience and being able to use the theoretical knowledge from the classes practically.

One is able to get great exposure from the mines and geological department especially as a mining and mineral processing engineering student.

# **REFERENCES**

1. Books, K. S., & Kowalski, B. R. (1994). Theory of analytical chemistry. *Analytical Chemistry*, *66*(15), 782A-791A.
2. Miller, J. C., & Miller, J. N. (1988). *Statistics for analytical chemistry.*
3. *Logbook*