A

PROJECT REPORT ON

"SOME PHYSICO – CHEMICAL ANALYSIS OF GUWAHATI WATER SAMPLE BY ION CHROMATOGRAPHY"

Carried out at

NATIONAL INSTITUTE OF HYDROLOGY (NIH), ROORKEE

Submitted in partial fulfillment of the requirements for the award of

The degree of
MASTER OF SCIENCE
IN
CHEMISTRY

(Commercial method of chemical analysis)

Of

GURUKULA KANGRI VISHWAVIDYALAYA, HARIDWAR

Submitted by

PRIYA YADAV

UNDER THE SUPERVISION OF

Dr. ABHA SHUKLA
(Assistant Professor)

DEPARTMENT OF CHEMISTRY
KANYA GURUKUL CAMPUS
GURUKULA KANGRI VISHWAVIDYALAYA
HARIDWAR-249407(U.K)
SESSION:- 2016-2017

ल विज्ञान संस्थान
STITUTE OF HYDROLOGY
: हिष्ठा मयोभुवः

: C. K. Jain
entist 'G' & Head
vironmental Hydrology Divison

राष्ट्रीय जलविज्ञान संस्थान
(जल संसाधन, नदी विकास और गंगा संरक्षण मंत्रालय)
जलविज्ञान भवन, रुड़की – 247 667, भारत

National Institute of Hydrology
(Ministry of Water Resources, RD & GR, Govt. of India)
Jalvigyan Bhawan, Roorkee - 247 667, INDIA

ISO 9001
CERTIFIED QUALITY MANAGEMENT SYSTEM
SWISSCERT
NABCB
QM 031

Phone : +91-1332-272860, 2492
Fax : +91-1332-272860, 27212
Email : ckj_1959@yahoo.co.in
ckjain@nih.ernet.in

Dated: March 28, 2017

TO WHOM IT MAY CONCERN

This is to certify that **Ms. Priya Yadav**, Student of M.Sc. (Chemistry),4th Semester, **Kanya Gurukul Campus of Gurukul Kangri Vishwavidyalaya, Haridwar** has carried out the Project Work on the topic **"Some Physico-Chemical Analysis of Guwahati Water Samples by Ion Chromatography"** during the period from 10th January, 2017 to 30th March, 2017 under my guidance and supervision.

She took full interest in her work and I sure that this training will help her in taking up related assignment in her career.

I wish her all success in life.

(C. K. Jain)

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Department of Chemistry
KANYA GURUKUL CAMPUS, HARIDWAR
Gurukul Kangri Vishwavidyalaya, Haridwar
(NAAC 'A' Grade Accredited Deemed To Be University u/s 3 of UGC Act 1956)

Ref. No. 167 Date 25/3/17

CERTIFICATE

This is to certify that Ms. **Priya Yadav** student of M.Sc., Chemistry, **(Commercial Methods of Chemical Analysis)**, **Kanya Gurukul Campus of Gurukul Kangri Vishwavidyalaya, Haridwar** has carried out her project work entitled **"Some Physio-Chemical Analysis of Guwahati Water samples by Ion chromatography "** under my supervision at **National Institute of Hydrology (NIH), Roorkee** for the partial fulfillment of the requirement for the award of the degree of Master of Science in Chemistry of Gurukul Kangri Vishwavidyalaya, Hardwar.

It is also certified that she has made pre submission presentation in the department.

Date :

Abha Shukla
(Dr. Abha Shukla)
Supervisor

P & T Office : Jwalapur - 249407, Haridwar (Uttarakhand) ☎ 01334-213522

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I owe my sincere thanks to my respected guide **Dr.C.K.Jain, Scientist 'G' and Head, Environmental Hydrology Division at NATIONAL INSTITUTE OF HYDROLOGY ROORKEE,** who allowed me to complete my project work and rendered his co-operation and help in preparing on the bases of practical. I am also grateful to the working staff of National Institute of Hyrdology. Who helped me at the time of my project work.

Lastly, I am thankful to my parents and family for being a constant source of inspiration.

Priyayadav.

Priya yadav

DECLARATION

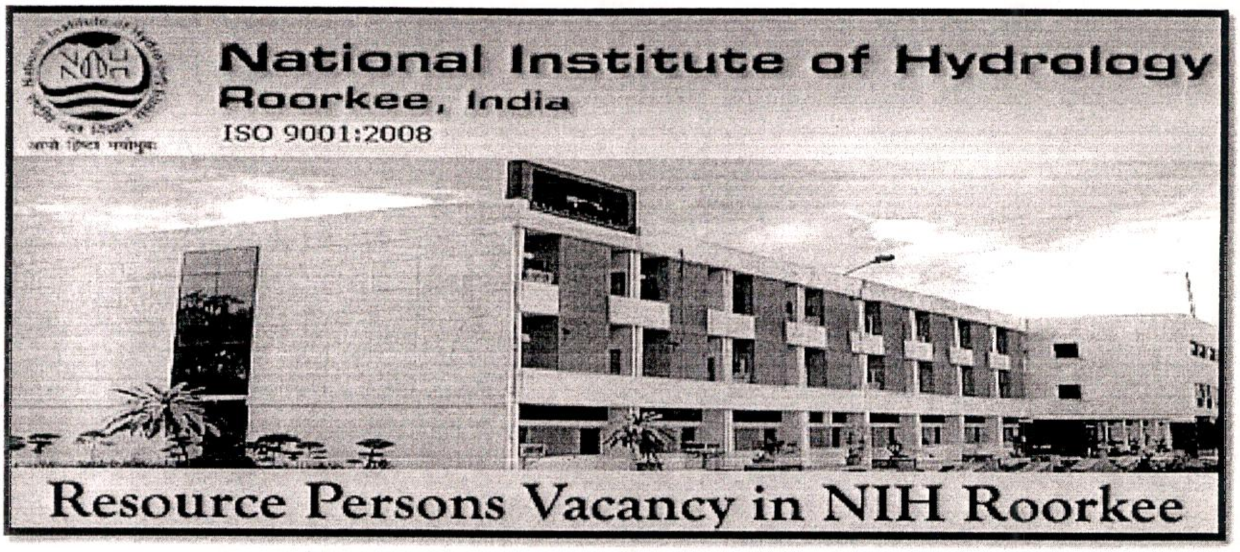
I here by declare that the work done in the project report entitled **"SOME PHYSICO – CHEMICAL ANALYSIS OF GUWAHATI WATER SAMPLES BY ION CHROMATOGRAPHY** "has been carried out by me during January to march 2017 at **NATIONAL INSTITUTE OF HYDROLOGY ROORKEE.**It is submitted in partial fulfillment of requirement for the award of Master's Degree in Chemistry (Commercial Methods of Chemical Analysis) of GurukulakangriVishwavidyalaya, Haridwar. It is an authentic record of my own work carried out under the guidance of Assistant Professor Abha Shukla, Department of Chemistry, Kanya Gurukula Campus, Jwalapur, Haridwar.

Priyayadav.

Date : 3|April|17

Priya Yadav

BRIEF OVERVIEW OF NIH



NATIONAL INSTITUTE OF HYDROLOGY,the premier institute in the area of hydrology and water resources in India. The institute was established in 1978 with the main objective of undertaking, aiding, promoting and co-ordinatingsystemating and scientific work in all aspect of hydrology. The institute is well equipped to carry out computer, laboratory and field oriented studies.

The institute is located at Roorkee in Haridwar District, Uttarkhand,India .Roorkee, the historic town, is a well known education and research centre with the I.I.T.Roorkee and a number of R & D organization viz. central building research institute, irrigation research institute and army's Bengal engineering group.The studies and research activities at the NIH Roorkee are carried out under five scientific division. As part of the technology transfer program of institute, various training courses/workshop are also organized by the division.The five division are as follow:

1.Hydrology division.

2.Groundwater hydrology division.

3.Hydrological investigations division

4.Surface waterhydrology division.

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INTRODUCTION-

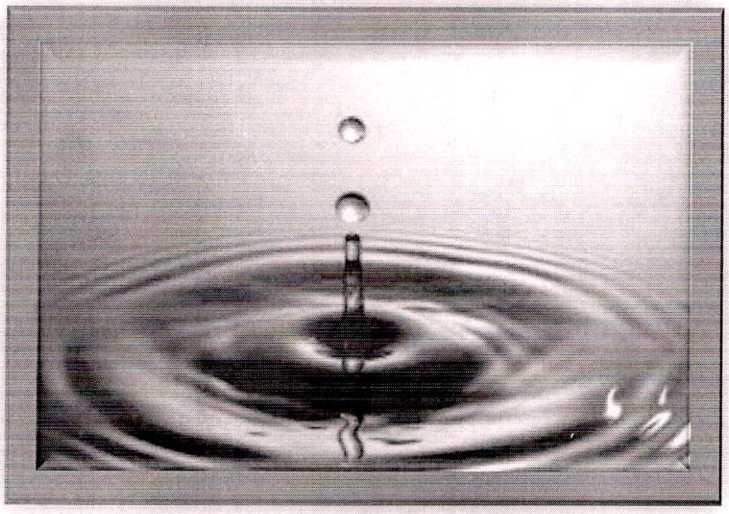
- WATER
- GROUND WATER
- WATER QUALITY
- WATER QUALITY ANALYSIS

Chapter-1

INTRODUCATION

Water is the most common liquid on Earth. It covers about 71.4% of the Earth. Pure water has no smell, taste, or colour. Lakes, oceans, and rivers are made of water. Precipitation is water that falls from clouds in the sky. It may be rain (liquid) if warm, or it may be frozen if cold. If water gets very cold (below 0 degrees Celsius), it freezes and becomes ice. If water gets very hot (above 100 degrees Celsius), it boils and becomes steam. Water is very important for life. However, some studies suggest that by 2025 more than half the people around the world will not have enough water.

Water is a fluid. Water is the only chemical substance on earth that exists naturally in three states. People know of over 40 anomalies about water. Unlike most other liquids such as alcohol or oil, when water freezes, it expands by about 9%. This expansion can cause pipes to break if the water inside them freezes.



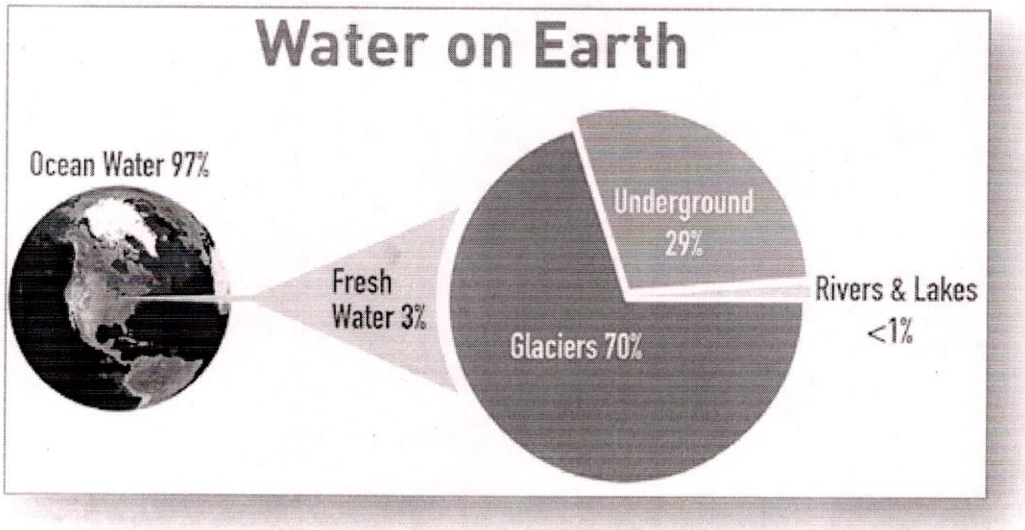
Water is a molecule made of 2 hydrogen atom and 1 oxygen atom. Its chemical formula is H_2O. Like other liquids, water has a surface tension, so a little water can make drops on a surface, rather than always spreading out to wet the surface. Water can also be called 'aqua', which is the Roman word for water.

Water is our most precious resource.All plant and animal must have water to survive. If there is no water there would be no life on earth .

The decreased mass of the shrinking glacier also spell water supply shortage in the near future. In fact, people living in the place near Himalayas are already facting water shortage .

Generally the fresh water on earth is locked up in glacier, icecaps,lakes,rivers ponds etc. India is fortunate to have abundant fresh water resource in the form of a number of perennial rivers beside reservoirs, ponds, lakes and swamps etc,but it is unfortunate that today most of our water bodies or fresh water resources are the most polluted and water from many of them is unfit for use . We use water for drinking, cooking, washing and growing our food as well as many other things.

Even water is used by industrial to generate electricity ,manufacture thingsand transport people and goods. Water is also essential in industry. It is heated and the steam is used to run machinery . Water is used to cool hot metal such as the production of steel.



The distribution of water on the Earth's surface is extremely uneven. Only 3% of water on the surface is fresh; the remaining 97% resides in the ocean. Of freshwater, 69% resides in glaciers, 30% underground, and less than 1% is located in lakes, rivers and swamps. Looked at another way, only one percent of the water on the Earth's surface is usable by humans, and 99% of the usable quantity is situated underground.

GROUNDWATER

Groundwater is the water present beneath Earth's surface in soil pore spaces and in the fractures of rock formations. A unit of rock or an unconsolidated deposit is called an aquifer when it can yield a usable quantity of water. The depth at which soil pore spaces or fractures and voids in rock become completely saturated with water is called the water table. Groundwater is recharged from, and eventually flows to, the surface naturally; natural discharge often occurs at springs and seeps, and can form oases or wetlands. Groundwater is also often withdrawn for agricultural, municipal, and industrial use by constructing and operating extraction wells. The study of the distribution and movement of groundwater is hydrogeology, also called groundwater hydrology.

Typically, groundwater is thought of as water flowing through shallow aquifers, but, in the technical sense, it can also contain soil moisture, permafrost (frozen soil), immobile water in very low permeability bedrock, and deep geothermal or oil formation water. Groundwater is hypothesized to provide lubrication that can possibly influence the movement of faults. It is likely that much of Earth's subsurface contains some water, which may be mixed with other fluids in some instances. Groundwater may not be confined only to Earth. The formation of some of the landforms observed on Mars may have been influenced by groundwater. There is also evidence that liquid water may also exist in the subsurface of Jupiter's moon Europa.

Groundwater is often cheaper, more convenient and less vulnerable to pollution than surface water. Therefore, it is commonly used for public water supplies. For example, groundwater provides the largest source of usable water storage in the United States, and California annually withdraws the largest amount of groundwater of all the states. Underground reservoirs contain far more water than the capacity of all surface reservoirs and lakes in the US, including the Great Lakes. Many municipal water supplies are derived solely from groundwater.

Iron and manganese: Depending on local condition ,ground water can be aeobic i.e. in the presence of oxygen gas,or anaerobic i.e. in the absence of oxygen – containing acceptors . In anaerobic conditions , iron and manganese containing minerals are relatively should and can dissolved into the water . When water is aerated and chlorinated , the iron and manganese react to form insoluble species and precipitate and cause rust and black – coloured stains or laundry and plumbing fixture .

Hardness: Hardness is a characteristic of water caused by the presence of calcium and magnesium , which are abundant in the earth 's crust . Hard water does not cause negative health impacts , but it reacts with soap to form a ppti.e soap scum , leaves water sports on surfaces , and forms precipitates in the water hearts , tea pots , heat exchanger , and pipes , reducing their efficiency .

Trace inorganic: Minerals can contain many trace elements ,including ,arsenic , barium , chromium , fluoride , selenium , and species that exhibit radioactivity such as radium , radon , and uranium . Many trace inorganic exhibit or other adverse health effects , if concentrations are too high .

Salinity: Brackish ground water with low to moderate salinity , ranging from about 1000 to 5000 mg/L total dissolved solids , is relatively common . Brackish water is too salty for potable , industrial or agricultural applications .

In addition to these natural constituents , ground water can cantain variety of anthropogenic contaminants .

WATER QUALITY

The quality of both surface and ground water is governed by a set of complex interacting physical , chemical and biological processes with reference to various beneficial uses of water . Traditionally human health as influenced by consumptive use of water as well as involving body contact (bathing and swimming) has been the primary concern .Although , many other beneficial uses of water such as fisheries , agriculture , industry and recreation have also been acknowledged.

In recent years , the concern for water quality has broadened in scope beyond humans and wildlife to the ecological integrity of surface water also . The management of water quality therefore requires an understanding of all the physico-chemical processes and the role of biota and the ecological processes as well as rehabilitation .

The term **water quality** is a widely used expression , which has an extremely broad spectrum of meanings .Each individual has vested interests in water for his particular use .The term water quality therefore , must be considered relative to the proposed use of water . From the user's point of view , the term **water quality** is defined as physical , chemical and biological characteristics of water by which is user evaluates the acceptability of water .

For example for the sake of man's health , we require that his water supply be pure , whole some and potable .In recent years the rivers are being used indiscriminately for disposal of municipal , industrial and agricultural wastes there by polluting the river water beyond the permissible limits . Due to this , the river water is gradually becoming unfit ever for irrigation purpose at some place . Thus it has become very essential to evaluate the environmental impacts of water resources to minimize the progressive deterioration in the quality of water .Therefore , a detailed study of water quality in all the vulnerable rivers is imperative for better management and use of water for different purposes.

MAJOR WATER QUALITY ISSUES

Pollution due to urbanization:

Urbanization has encouraged the migration of people from villages to the urban areas . This has given rise to the number of environmental problems such as water supply , waste water generation and its collection , treatment and disposal .In urban areas , water is tapped for domestic and industrial use from rivers , streams , wells and lake . Approximately 80% of the water supplied for domestic use passes out as waste water . In most of the case , waste water is let out untreated and it either percolates in to the ground in turn contaminates the ground water or is discharged into the natural drainage system causing pollution in downstream areas .

Eutrophication:

The discharge of domestic waste water , agricultural return water or runoff water and many industrial effluents contribute nutrients like phosphate and nitrates . These nutrients promote excess growth of algae in the water bodies . This is not desirable for balance aquatic ecosystem .

Salinity:

Salinity is increasing in many water bodies especially ground water due to leaching of salts build-up in agricultural areas under intense irrigation . A number of industrial activities discharge waste water with high dissolved solids causes increase in salinity of water .Presence of toxic substance impairs the water quality by making it unfit for human consumption , aquatic life and irrigation .

Natural contaminants:

By far the greats water quality problem in developing countries is the prevalence of water borne diseases . Yet in , addition of the human induced pollution problems , water supply also has specific natural quality problems , which are mainly related to local geology. Some of the specific problems of the natural origin includes fluoride , arsenic etc .

Pathogenic Pollution:

Water borne diseases are the most important water quality issues in India . These is mainly due to inadequate arrangements for transport and treatment of waste water . A major portion of the waste water generated from human settlements is not properly transported and treated before discharging into natural waters . This results in contamination of both surface and ground waters .More over , contribution of pathogens through dissfuses source is also quite significant . Thus , most of the surface water bodies and many ground water sources are contaminated .

A large population of the country still uses water directly for drinking or contact use with outtreatment , thus being exposed to the water borne diseases . This is the single major cause for mortality due to water pollution .

Ecological Health:

A large number of areas in our aquatic environment support rare species and ecologically very sensitive . They need special protection . Since , the water act 1974 provide for maintenance and restoration of wholesomeness of aquatic resources , which is directly related to ecological health of the water bodies , it is important that ecological health of the water bodies is given first priority in the water quality goal .

WATER QUALITY ANALYSIS

Water quality refer to the chemical , physical , biological and radiological characterstic of water . It is a measure of the condition of water relative to the requirements of one and more biotic species and or to any human need or purpose . It is most frequently used by reference to a set of standards against which compliance can be assessed . The most common standards used to assess water quality relate to health of ecosystems , safety of human contact and drinking water. Therefore , it is used to assess the physical , chemical and biological characteristics of water .

Water quality analysis including following parameters :

. PHYSICAL PARAMETERS

. CHEMICAL PARAMETERS

. BIOLOGICAL PARAMETERS

PHYSICAL CHARACTERISTICS OF WATER :

1. **Smell / order**
2. **Taste**
3. **Colour**
4. **Solids**
5. **Turbidity**
6. **Temperature**
7. **Conductivity**

CHEMICAL CHARACTERISTICS OF WATER :

1. Hardness
2. Alkalinity
3. pH
4. Nitrate
5. Nitrite
6. B.O.D.
7. C.O.D.
8. D.O.
9. Major Cations (Na^+ , K^+ , Ca^{2+} , Mg^{2+})
10. Major Anions (Cl^- , SO_4^{2-}, PO_4^{3-}, HCO_3^-)

CHAPTER-2

- **PURPOSE OF STUDY**

chapter -2

PURPOSE OF THE SYUDY

In India 12% of people get clean drinking water , the rest 88% quench their thirst from polluted lakes , tanks , rivers and wells due to which more than three million people get affected or die from enteric diseases every year .The water borne diseases are jaundice , cholera , typhoid and gastro enteritis etc . This surface water and groundwater is mainly polluted by anthropogenic activities viz. urbanization , industrialization , disposing garbage etc.

Water quality of lakes is deteriorating due to solid and liquid disposal of wastes .Lakes , being stagnant water bodies , are more prone to pollution than the rivers as in lakes self purification process are less effective than rivers . Any contamination or pollution of lakes effects greatly the flora and fauna and also the human health if the water is used for domestic supply . The environmental health of any lake system depends upon the nature of that lake and its exposure various environmental factors such as temperature , depth of water , wind speed , soil types and land uses of the catchment .

Groundwater level is declining and also groundwater contamination is reported in various part of India especially in shallow aquifer. Hence , Guwahati , Assam , has been selected for ground water quality assessment for suitability of drinking purpose .

Six samples of Guwahati ground water has been analyzed for determine the anion that is fluoride , chloride , nitrate , sulphate and cation that is sodium , potassium , calcium and magnesium etc. In Guwahati ground water samples by using ion chromatography . And also determination the water quality parameters viz. pH , Electrical Conductance , Alkalinity and hardness .

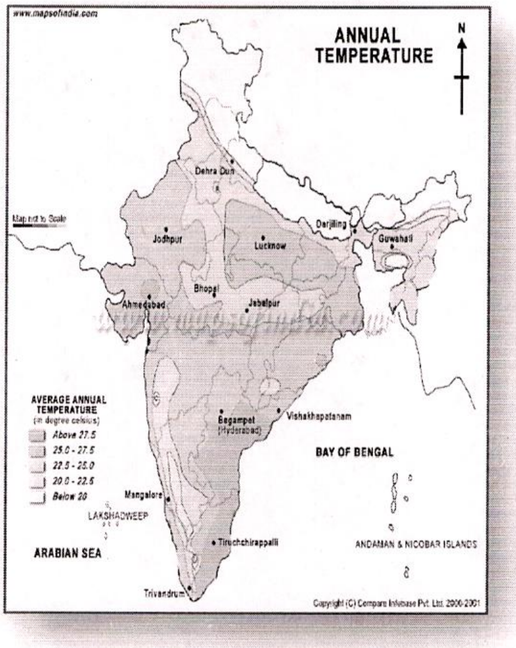
CHAPTER-3

- STUDY AREA
- SAMPLING AND PRESERVATION TECHNIQUES
- PHYSICO-CHEMICAL ANALYSIS
- AUTO-TITRATION

Chapter – 3

EXPRIMENTAL WORK

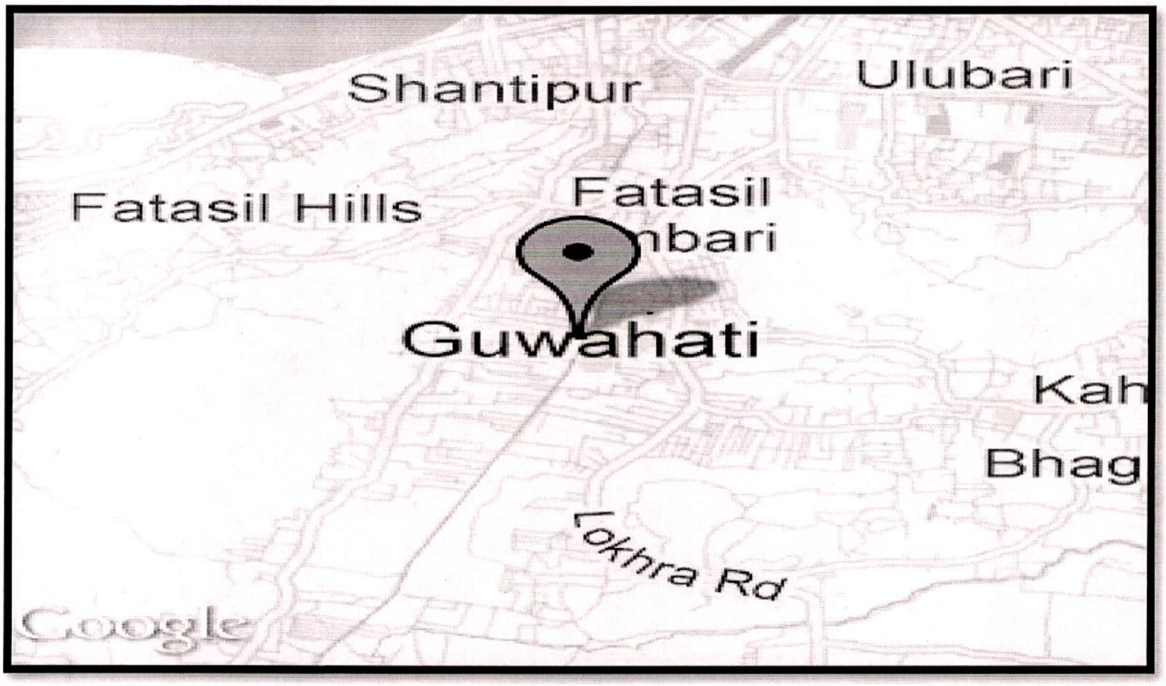
study Area



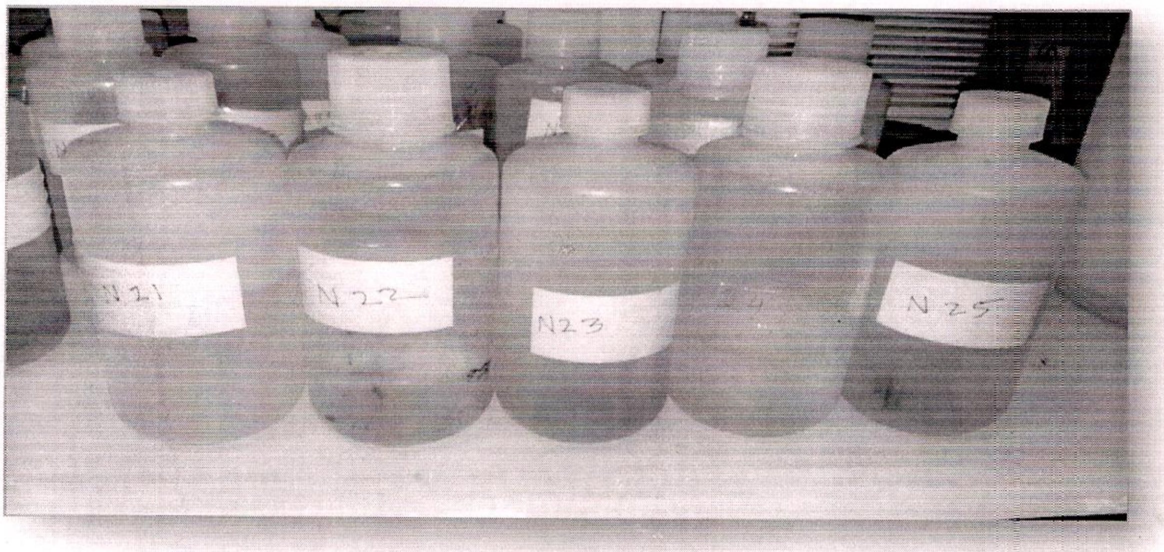
Guwahati is the largest city of Assam and Northeastern India, a major riverine port city and one of the fastest growing cities in India, situated on the South Bank of the Brahmaputra River.

The ancient cities of Pragjyotishpura and Durjaya (North Guwahati) were the capitals of the ancient state of Kamarupa under the Varman and Pala dynasties. Many ancient Hindu temples are in the city, giving it the name "City of Temples". Dispur, the capital of Assam, is in the circuit city region located within Guwahati and is the seat of the Government of Assam.

Guwahati lies between the banks of the Brahmaputra River and the foothills of the Shillong plateau, with LGB International Airport to the west and the townof Narengi to the east. It is gradually being expanded as North Guwahati to the northern bank of the Brahmaputra. The noted MadanKamdev is situated 30 kilometres (19 mi) from Guwahati. The Guwahati Municipal Corporation (GMC), the city's local government, administers an area of 216 square kilometres (83 sq mi), while the Guwahati Metropolitan Development Authority (GMDA) is the planning and development body of greater Guwahati Metropolitan Area. With an area of 1,528 square kilometres (590 sq mi), Guwahati is the second-largest metropolitan region in eastern India, after Kolkata .



SAMPLING AND PRESERVATION TECHNIQUES



Sampling is the first step leading to the generation of water quality data and is an exceedingly important ; care must be taken to ensure obtaining a sampie that is truly representative thoroughly cleaned plastic or glass bottles fitted with screw caps may be used for water sample collected with depth integrating samplers , plastic container are generally preferred for inorganic samples and glass is preferred for organic samples .

Compact system for routine analysis of anion ,cations and polar substances are in the range of µgm/ L to gm/L . The samples of water were collected weekly at 8:30 am on every Sunday . The displacement between each sampling site is 2 km (approx.) .

Determination of pH should be done in the field . Sample for metal analysis can be preserved by addition of Nitric acid , sample for organic constituent 's determination by chilling or freezing . A list containing method of sample preservation and time allowed between sample collection and analysis is presented in following table:

TABLE 3.2 Sample location and Identity

S.NO.	SAMPLE SITE	SAMPLE ID	DATE	VOLUME OF SAMPLE
1.	GUWAHATI	N-21	25-01-2017	1 LITRE
2.	GUWAHATI	N-22	25-01-2017	1 LITRE
3.	GUWAHATI	N-23	25-01-2017	1 LITRE
4.	GUWAHATI	N-24	25-01-2017	1 LITRE
5.	GUWAHATI	N-25	25-01-2017	1 LITRE
6.	GUWAHATI	N-26	25-01-2017	1 LITRE

TABLE 3.2 Adopted preservation technique

Parameter	Preservation	Maximum storage time
Colour	Cool to 3-4°C	24 hours
Turbidity	Cool to 3-4°C	7 days
pH		Immediately
Alkalinity	Cool to 3-4°C	24 hours
Hardness	Add 2ml HNO_3 /L Cool to 3-4°C	7 days
Sulphate	Cool to 3-4 °C	7 days
Chloride		7 days
Phosphate	Cool to 3-4°C	7 days
Nitrogen,Nitrate	Add 2ml H_2SO_4/L , Cool to 3-4 °C	7 days

PHYSICO – CHEMICAL ANALYSIS:

1.ElectricalConductivity

2.pH

3. Alkalinity

4. Hardness

Electrical Conductivity

Conductivity is the measure of capacity of a substance or solution to conduct electric current . Conductivity is reciprocal of the resistance . Conductivity if measured of a cube with each side of 1 cm at 25^0 C is called specific conductance . As most of the salts in the water are present in the ionic forms , capable of conducting current , therefore , conductivity is a good and rapid measured of the total dissolved solid . As the dissolved solids in the highly mineralized waters are usually more than 65% of the conductivity , the value of dissolved solids , as a general rule , can be obtained by multiplying it by a factor of 0.65 .

The conductivity of distilled water ranges between 1-5µ mho but the present of salts and contamination with waste waters increases the conductivity of water .consequently , a sudden rise in conductivity in the water will indicate addition of some pollutans to it .

The conductivity of generally reported in mmho or µmho. The recent unit of conductivity has been named as Siemens(S) instead of mho.

PRINCIPLE

Electrical conductance is the ability of a substance to conduct the electric current. In water , it is the property caused by the presence of various ion species.

It is generally measured with the help of a conductivity meter having conductance cell containing electrodes of platinum coated with Pt black carbon .Conductance ,when measured between the electrodes having a surface area of 1cm2 and placed at a distance of a 1cm,is called electrical conductivity and is the property of the water sample, rather than the measuring system. The term specific conductance is also used in place of electrical conductivity .

The unit of conductivity is siemens**(S) Cm**$^{-1}$.The conductivity of most water is generally low so the low so the unit **µS Cm**$^{-1}$ shall be much appropriate .

pH

pH is the measure of the intensity of acidity or alkalinity and measures the concentration of hydrogen ions in water . It does not measure total acidity or alkalinity .Infact , the normal acidity or alkalinity depends upon excess of H^+or OH^- ions over the other , and measured in normality or gram equivalents of acids or alkali . If free H^+ are more than OH^-ions , the water shall be acidic , or alkaline the other way round .

pH is generally measured on a Log scale and equals to negative $\log_{10}$ of hydrogen ion concentration .

$$\mathbf{pH = -\log_{10}[H^+]}$$

$$\mathbf{= \log_{10} 1/[H^+]}$$

pH scale ranges from 0 – 14 with 7 as neutral , below 7 being acid and above 7 as alkaline .

PRINCIPLE

The pH can be defined as a negative logarithum of hydrogen ion concentration expressed molarity . The pH scale is series of number that express the degree of acidity or alkalinity of a solution . The pH scale ranges from 0 – 14 with 7 as neutral , below 7 being acid and above 7 as alkaline .

ALKALINITY

Alkalinity in natural water is due to free hydroxyl ions and hydrolysis of salts formed by weak acids and strong bases .

$$A^- + HOH = HA + OH^-$$

When a salt of weak acid and strong base is hydrolysed . The number of milliequivalents of acid used in the titration of combine all the hydroxyl ions is called a as total alkalinity .

$$CO_2 + H_2O \leftrightharpoons H_2CO_3$$

$$H_2CO_3 \leftrightharpoons H^+ + HCO^-_3$$

$$HCO_3^- \leftrightharpoons H^+ + CO_3^{2-}$$

$$CO_3^{2-} + 2HOH \leftrightharpoons H_2CO_3 + 2OH^-$$

$$HCO_3^- + HOH \leftrightharpoons H_2CO_3 + OH^-$$

Mostly of the alkalinity in natural waters is formed due to dissolution of CO_2 In water . Carbonate and bicarbonate thus formed are dissociated to yield hydroxyl ions . Carbonate salts produce double the hydroxyl ions than the bicarbonates . Alkalinity is also prodused by the action of water on limestone or chalk .

$$CaCO_3 + H_2O + CO_2 \leftrightharpoons Ca(HCO_3)_2$$

PRINCIPLE

Total alkalinity is the measure of the capacity of the water to neutralize a strong acid . The alkalinity in the water is generally imparted by the salts of carbonates , bicarbonates , phosphates ,nitrates , borates , silicates , etc. Are rich in carbonates and bicarbonates with little concentration of other alkalinity imparting ions .

Total alkalinity , carbonates and bicarbonates can be estimated by titrating the sample with a strong acid (HCl or H_2SO_4) , first to pH 8.3 using phenolphthalein as an indicator and then further to pH between 4.2 and 5.4 with methyl orange or mixed indicator . In first case , the value is called as phenolphthalein alkalinity (PA) and in second case , it is total alkalinity (TA) .

HARDNESS

Hardness is associated with capacity of water to precipitate soap or it is the property which reduced the lather formation with soap and it also increase the boiling point of the water . Temporary hardness is due to the presence of bicarbonate of Ca^{2+} and Mg^{2+} which can be remove only by prolonged boiling and bicarbonate of Ca and Mg are precipitated as normal carbonate by less of CO_2 on heating .

Total hardness is defined as the sum of the calcium and magnesium concentration , both expressed as $CaCO_3$, in mg/ L . The degree of hardness of drinking water has been classified in terms of the equivalent $CaCO_3$ concentration as follows :

Soft 0-60 mg /L

Medium 60-120 mg/L

Hard 120 – 180 mg/L

Very hard >180 mg/L

PRINCIPLE

Hardness is generally caused by calcium and magnesium ions present in water. Polyvalent ions of some other metals like strontium, iron,aluminium, zinc and manganese etc. are also capable of precipitating the soap and thus contributing to the hardness. However, the concentration of these ions is very low in natural water, therefore, hardness is generally measured as concentration of only calcium and magnesium, as calcium carbonate,which are far higher in quantities over other hardness producing ions .

Calcium and magnesium from a complex of wine red colour with Eriochrome black – T at pH 10.0 ±0.01. The EDTA had got a stronger affinity towards Ca^{++} and Mg^{++} and, therefore, by addition of EDTA, the former complex is broken down and a new complex of a blue colour is formed.

$$M^{++} + \text{E.B.T.} \longrightarrow M^{++}(\text{E.B.T.})$$

Wine red colour

$$M^{++} + \text{EDTA} \longrightarrow M^{++}(\text{EDTA})$$

Blue colour

CALCULATION

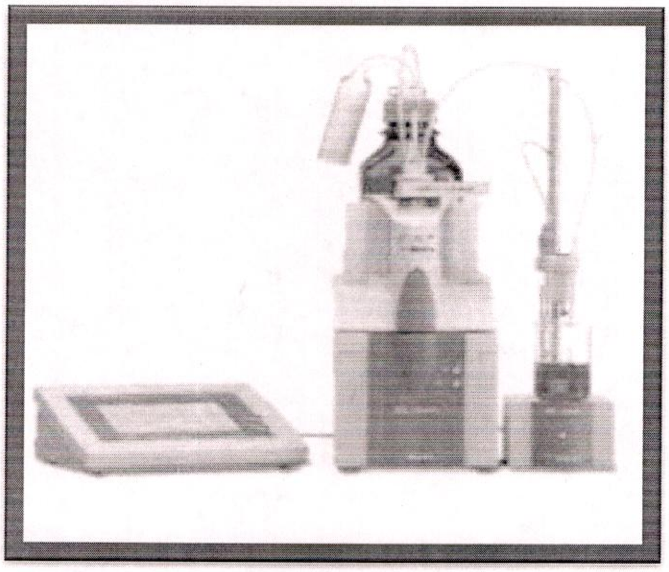
Ca^{2+} hardness =Ca^{2+} concentration (mg/ L)/0.401

Mg^{2+} hardness = Mg^{2+} concentration (mg/L)/0.243

Total hardness =(Ca^{2+} + Mg^{2+}) hardness

pH , Alkalinity is measured by

AUTO TITRATION



A titration is a technique where a solution of known concentration is use to determine the concentration of an unknown solution. Typically, the titrant (the known solution) is added from a burette to a known quantity of the analyte (the unknown solution) until the reaction is complete .

The whole process of titration is made much easier by making it automated.we simply add a predetermined amount of reactant and the machine will add the other reactant and measure the products to find the end point . Many samples can be done in very short time. The accuracy is increased due to the finely calibrated computer instead of our eyes. The amount of hands on interaction is drastically reduced. The Auto- titration was carried out in the laboratory using **888-TITRANDO METROHM.**

AUTOTITRATOR

Automated titrators are micro-processor controlled instruments which allow the automation of all operations involved in titration:

- **Titrant addition**
- **Monitoring of the reaction**
- **Recognition of the end point**
- **Data storage**
- **Calculation**
- **Result storage**
- **Transfer of data to printer or computer**

In addition to the automatic titrator can be potentiometric titration , including acid-base titration , redox titration but can also be a constant pH measurements when the automatic titrator and automatic sample changer multi line use , not only can efficiently automate the measurement of large number of samples , but also improve the analysis of repeatability , reliability, easy operation, saving time and effort . suitable for quality control , inspection , analysis all aspects of research , development and so on .

CHAPTER-4

- ION CHROMATOGRAPHY
- INSTRUMENTATION
- ADVANTAGES
- DISADVANTAGES

History of ion chromatrography

The ion chromatography primarily began between 1935–1950 during World War II and it was through the "Manhattan project" that applications and IC were significantly extended. Ion chromatography was originally introduced by two English researchers, agricultural Sir Thompson and chemist J T Way. The works of Thompson and Way involved the action of water-soluble fertilizer salts, ammonium sulfate and potassium chloride. These salts could not easily be extracted from the ground due to the rain. They performed ion methods to treat clays with the salts, resulting in the extraction of ammonia in addition to the release of calcium. It was in the fifties and sixties that theoretical models were developed for IC for further understanding and it was not until the seventies that continuous detectors were utilized, paving the path for the development from low-pressure to high-performance chromatography. Not until 1975 was "ion chromatography" established as a name in reference to the techniques, and was thereafter used as a name for marketing purposes. Today IC is important for investigating aqueous systems, such as drinking water. It is a popular method for analyzing anionic elements or complexes that help solve environmentally relevant problems . Likewise, it also has great uses in the semiconductorindustry .

Chapter-4

ION CHROMATOGRAPHY

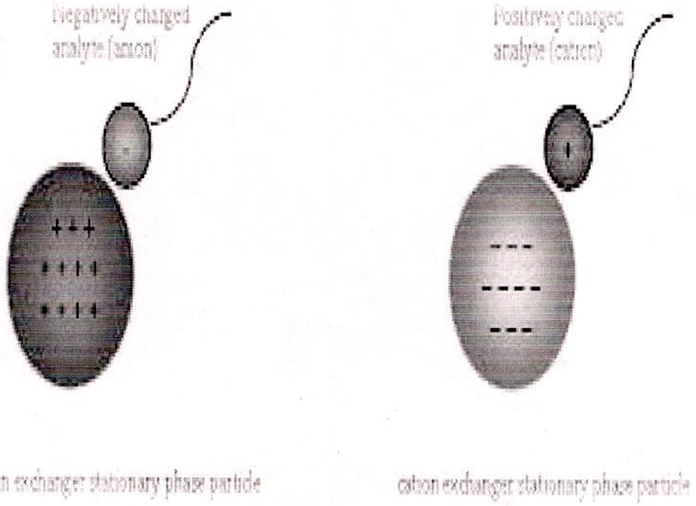
INRODUCTION

Ion chromatography (or **ion-exchange chromatography**) is a chromatography process that separates ions and polar molecules based on their affinity to the ion exchanger. It works on almost any kind of charged molecule—including large proteins, small nucleotides, and amino acids. It is often used in protein purification, water analysis, and quality control. The water-soluble and charged molecules such as proteins, amino acids, and peptides bind to moieties which are oppositely charged by forming ionic bonds to the insoluble stationary phase.

The equilibrated stationary phase consists of an ionizable functional group where the targeted molecules of a mixture to be separated and quantified can bind while passing through the column—a cationic stationary phase is used to separate anions and an anionic stationary phase is used to separate cations. Cation exchange chromatography is used when the desired molecules to separate are cations and anion exchange chromatography is used to separate anions.

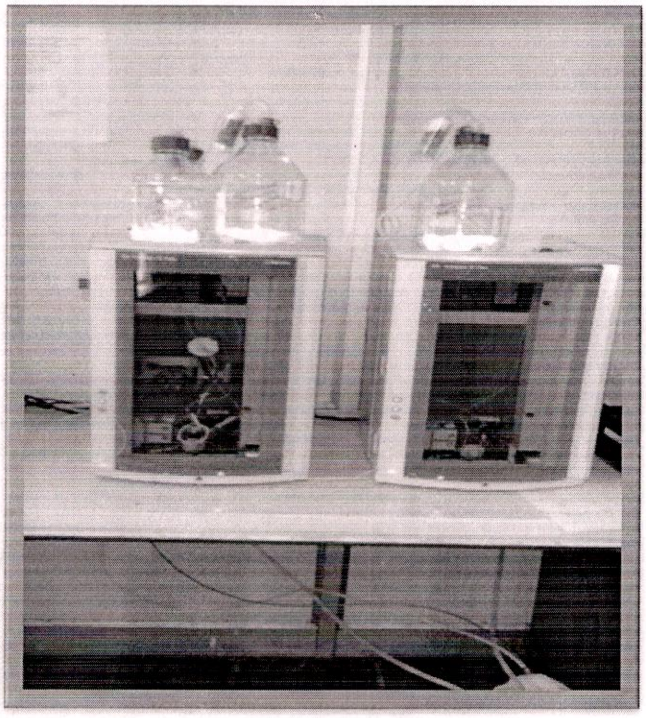
The bound molecules then can be eluted and collected using an eluant which contains anions and cations by running higher concentration of ions through the column or changing pH of the column. One of the primary advantages for the use of ion chromatography is only one interaction involved during the separation as opposed to other separation techniques; therefore, ion chromatography may have higher matrix tolerance. However, there are also disadvantages involved when performing ion-exchange chromatography, such as constant evolution with the technique which leads to the inconsistency from column to column .

PRINCIPLE



Ion-exchange chromatography separates molecules based on theirrespective charged groups. Ion-exchange chromatography retains analyte molecules on the column based on coulombic (ionic) interactions. Essentially, molecules undergo electrostatic interactions with opposite charges on the stationary phase matrix. The stationary phase consists of an immobile matrix that contains charged ionizable functional groups or ligands. The stationary phase surface displays ionic functional groups (R-X) that interact with analyte ions of opposite charge. To achieve electroneutrality, these inert charges couple with exchangeable counterions in the solution. Ionizable molecules that are to be purified compete with these exchangeable counterions for binding to the immobilized charges onthe stationary phase. These ionizable molecules are retained or eluted based on their charge.

PREPARTION OF MOBILE PHASE



CATION –Take 284mg di piclonic acid in distilled water and heat to dissolve then cool and add 1.7ml of HNO_3 and make up to 1 litre with distilled water .

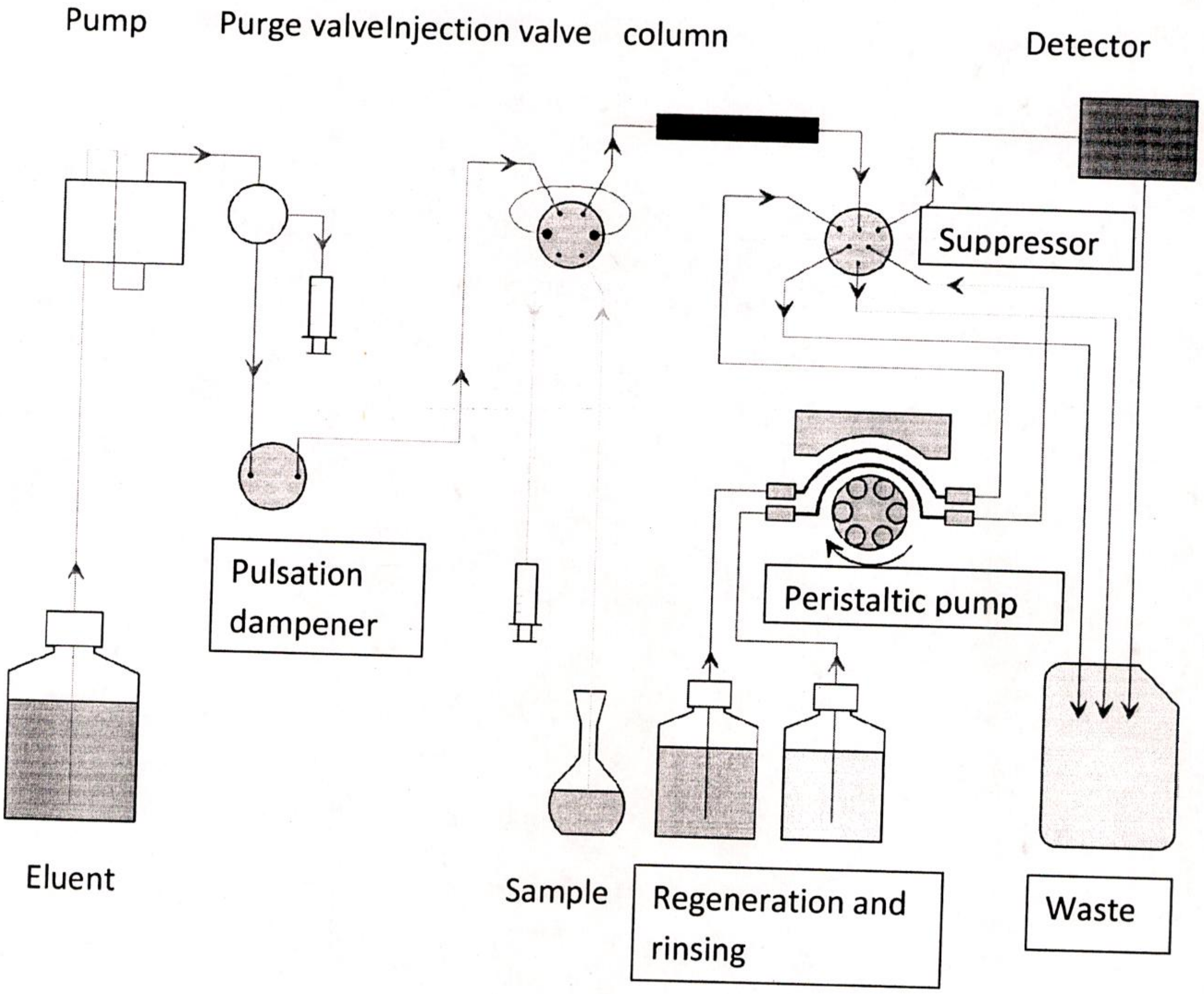
ANION – Take 339mg of sodium carbonate and 84mg of sodium bicarbonate dissolve in distilled water and make up to 1 litre .

FOR TITRATION - 0.01 M H_2SO_4

ION CHROMATOGRAPY PROCESS

First of all the sample is introduced then flows through the guard and into the analytical ion exchange columns where the ion exchange occurs . After separation, the suppressor reduces the conductivity of the eluent and increase the conductivity of the analytes . So they are delivered to the detector . A computer and software are used to control the system .

INSTRUMENTATION



Ion chromatography consist of following components-

- **PUMP**
- **INJECTOR**
- **COLUMN**
- **SUPPRESSOR**
- **DETECTOR**
- **RECORDER**

PUMP-

The ion chromatography pump is considered to be one of the most important components in the system which has to provide a continuous constant flow of the eluent through the ion chromatography injector, column and detector.

The solvent then passes from the selector to a high pressure pump. The mobile phase passes form the pump to the sampling device, usually a sample rotating valve that on rotation places the sample in the line with the mobile flow which then passes on to the column.

INJECTOR-

Sample introduction can be accomplished in the various ways. The simplest method is to use an injection valve. In more sophisticated LC, automatic sampling devices are incorporated where sample introduction is done with the help of auto samplers and microprocessors. Injector should provide the possibility of injecting the liquid sample.

COLUMN-

Ion exchange column are vary widely in size, packing material and material opf construction. The column where the components of interest are separated. The column material may be stainless steel, titanium, glass or plastic. The life of a column will depend largely on the type of samples it is used to separate.

SUPPRESSOR-

Ion chromatography suppressor are membrane based devices. Suppressor can be used with the aqueous /organic eluents needed to elute organic analytes which are retained on the stationary phase.

The suppressor reduces the background conductivity of the chemical used to elute samples from the ion exchange column. Which improves the conductivity measurement of the ions being tested.

DETECTOR-

In ion chromatography the electrical conductivity is commonly used.An electricalfield is applied between the electrodes. Detector measures the electrical resistivity of ions in the solution. Conductivity is the reciprocal of resistance.

AMPLIFIER-

The amplifier output is then either digitized, and the binary number sent to a computer for storage and processing or the output is passed directly to a potentiometric recorder.

ADVANTAGES AND DISADVANTAGES OF ION CHROMATOGRAPHY

ADVANTAGES

- The main advantage of ion chromatography is that multiple ions can be analyzed at once since each ion elutes at different rate .Further more , the time it takes to analyse a sample is typically around 10 minutes .
- Shorter analysis time for simpler sample (generally 10-12 minute)
- small sample volume
- Long life of resins
- Cheap maintenance
- Metal –free peak system
- Powerful separation
- Ion chromatography developed into a powerful analytical technique versatility , speed of operation and reasonable cost are some of the factors that have contributed to its
- Usage of cheap , safe and environmental friendly chemicals.
- Highly selectivity for the detection of compounds like- halogens , peroxides , quinines , nitrates etc .

DISADVANTAGES

- Soil , sediment , geological sample prepration is more extensive .
- Longer analysis time for more complex samples .
- Can’t select specific compounds for high speed analysis .
- Some time overlap can occur in chromatogram when compounds have similar separation characteristics .
- Least sensitive to compounds whose molecules have negligible affinity for electron .
- Carrier gas used should be of pure from like- pure nitrogen.

CHAPTER-5

- RESULTS AND DISCUSSION
- REFERENCES

RESULTS:

The following results were obtained after the water quality analysis of Guwahati ground water:

TABLE – RESULTS OF PHYSIO CHEMICAL CHARATERISTICS OF VARIOUS SAMPLES:

S.NO.	SAMPLE ID	pH	HARDNESS	ALKALINITY
1.	N-21	8.2	68.587	135.200
2.	N-22	7.1	157.826	173.343
3.	N-23	7.6	144.897	154.457
4.	N-24	7.0 2	53.076	n.d.
5.	N-25	7.2	91.611	136.657
6.	N-26	7.3	131.634	156.614

TABLE – RESULTS OF CONCENTRATIONS OF ANIONS IN VARIOUS WATER SAMPLES OF GUWAHATI GROUND WATER:

ANIONS:

SAMPLE ID	FLUORIDE Mg/L	CHLORIDE Mg/L	NITRITE Mg/L	NITRATE Mg/L	SULPHATE Mg/L
N-21	0.324	1.006	–	0.136	6.623
N-22	0.125	23.920	–	1.172	8.198
N-23	0.289	1.843		0.339	0.077
N-24					
N-25	0.286	3.827	0.017	0.055	0.137
N-26	0.369	1.188	0.124	3.940	0.095

TABLE –RESULTS OF CONCENTRATIONS OF CATIONS IN VARIOUS WATER SAMPLES OF GUWAHATI GROUND WATER:

CATIONS:

SAMPLE ID	SODIUM Mg/L	AMMONIUM Mg/L	POTASSIUM Mg/L	CALCIUM Mg/L	MAGNESIUM Mg/L
N-21	33.547	–	1.211	18.975	5.163
N-22	25.118	–	4.226	37.231	15.781
N-23	6.837	0.107	1.567	46.565	6.978
N-24					
N-25	18.925	4.570	1.690	22.822	8.426
N-26	15.743	0.259	1.154	30.339	13.595

NIH IC REPORT

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N-21

Anion

Component name	Retention time min	Height µS/cm	Area (µS/cm) x min	Concentration mg/L
Fluoride	5.81	0.308	0.062	0.324
Chloride	9.12	0.538	0.123	1.006
Nitrate	16.84	0.018	0.009	0.136
Sulfate	27.40	0.960	0.575	6.623

Cation

Component name	Retention time min	Height µS/cm	Area (µS/cm) x min	Concentration mg/L
Sodium	12.00	12.698	3.758	33.547
Potassium	22.39	0.122	0.065	1.211
Calcium	28.55	2.019	1.516	18.975
Magnesium	41.23	0.977	0.916	5.163

% Error---------------------------------------.-0.478
Sample pH---------------------------------------8.2
M-alkalinitv as CaCO3----------------------------135.200
P-alkalinity as CaCO3----------------------------0.000
Total Hardness by IC-----------------------------68.587

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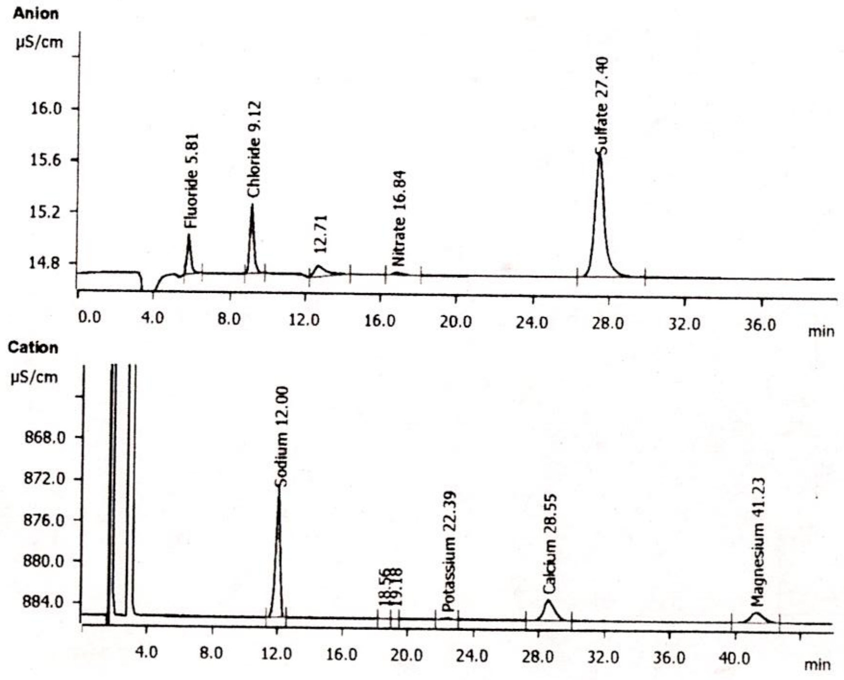

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N-22

Anion

Component name	Retention time	Height	Area	Concentration
	min	µS/cm	(µS/cm) x min	mg/L
Fluoride	5.81	0.044	0.010	0.125
Chloride	9.11	5.100	1.172	23.920
Nitrate	16.77	0.070	0.032	1.172
Sulfate	27.44	0.475	0.285	8.198

Cation

Component name	Retention time	Height	Area	Concentration
	min	µS/cm	(µS/cm) x min	mg/L
Sodium	11.94	4.047	1.125	25.118
Potassium	22.31	0.164	0.090	4.226
Calcium	28.68	1.615	1.190	37.231
Magnesium	41.13	1.176	1.120	15.781

% Error-------------------------------------.0.206
Sample pH-------------------------------------7.1
M-alkalinity as CaCO3--------------------------173.343
P-alkalinity as CaCO3--------------------------0.000
Total Hardness by IC---------------------------157.826

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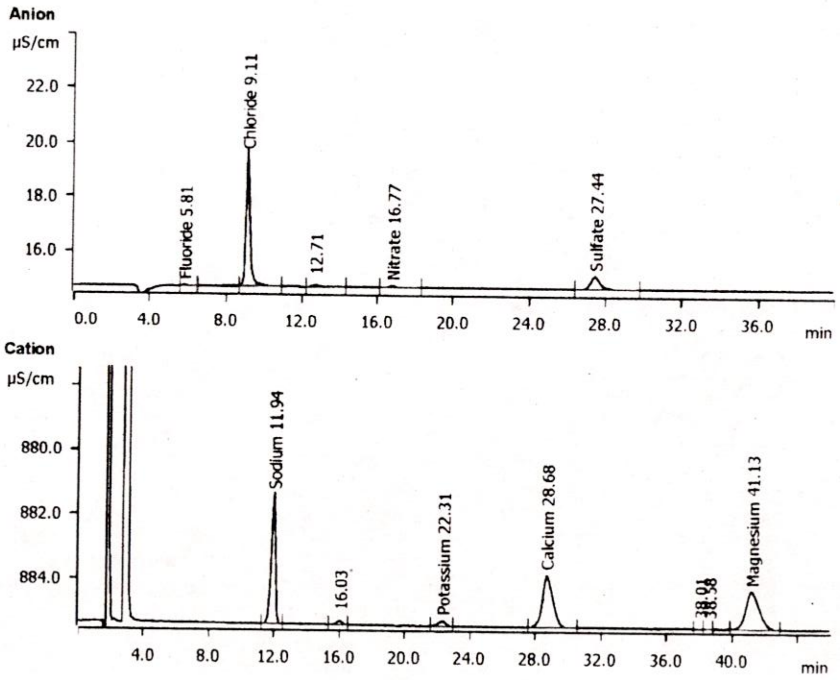

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N-23

Anion

Component name	Retention time min	Height µS/cm	Area (µS/cm) x min	Concentration mg/L
Fluoride	5.82	0.273	0.055	0.289
Chloride	9.10	0.949	0.226	1.843
Nitrate	16.73	0.049	0.023	0.339
Sulfate	28.02	0.014	0.007	0.077

Cation

Component name	Retention time min	Height µS/cm	Area (µS/cm) x min	Concentration mg/L
Sodium	11.91	2.784	0.766	6.837
Ammonium	13.96	0.015	0.005	0.107
Potassium	22.23	0.148	0.084	1.567
Calcium	28.12	4.181	3.720	46.565
Magnesium	41.06	1.308	1.238	6.978

% Error---------------------------------------.1.253
Sample pH---------------------------------------7.6
M-alkalinity as CaCO3---------------------------154.457
P-alkalinity as CaCO3---------------------------0.000
Total Hardness by IC----------------------------144.897

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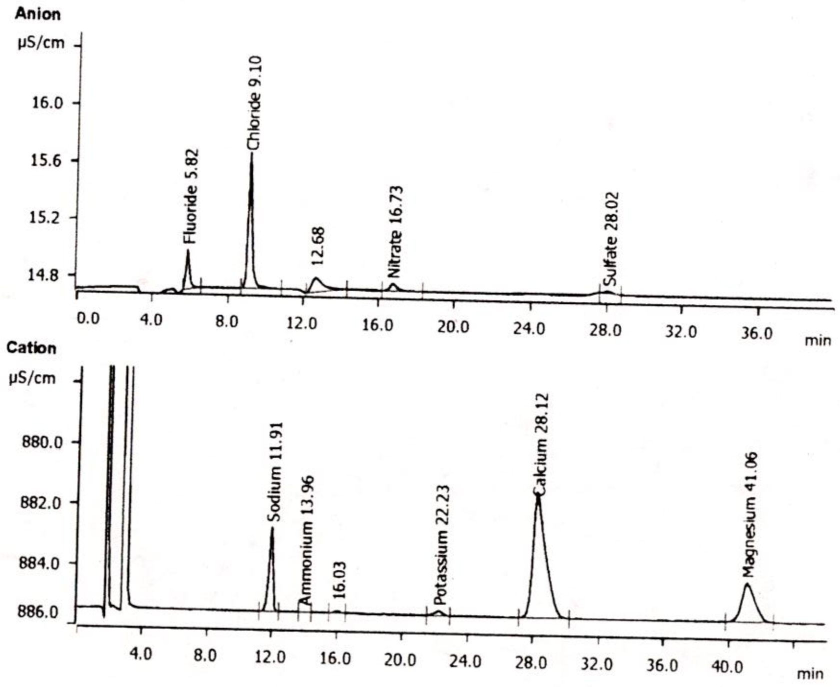

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N-24

Anion

Component name	Retention time min	Height µS/cm	Area (µS/cm) x min	Concentration mg/L
Fluoride	5.79	0.837	0.181	0.477
Chloride	9.09	0.537	0.126	0.516
Nitrite	11.23	0.007	0.002	0.015
Nitrate	16.60	2.037	0.863	6.244
Sulfate	27.20	0.027	0.013	0.076

Cation

Component name	Retention time min	Height µS/cm	Area (µS/cm) x min	Concentration mg/L
Sodium	11.89	11.295	3.279	14.636
Ammonium	13.88	0.037	0.013	0.128
Potassium	22.15	0.440	0.243	2.271
Calcium	28.13	2.397	1.813	11.345
Magnesium	39.97	2.094	2.156	6.078

% Error--.0.555

Sample pH--------------------------------------7.2

M-alkalinity as CaCO3----------------------------80.271

P-alkalinity as CaCO3----------------------------0.000

Total Hardness by IC-----------------------------53.312

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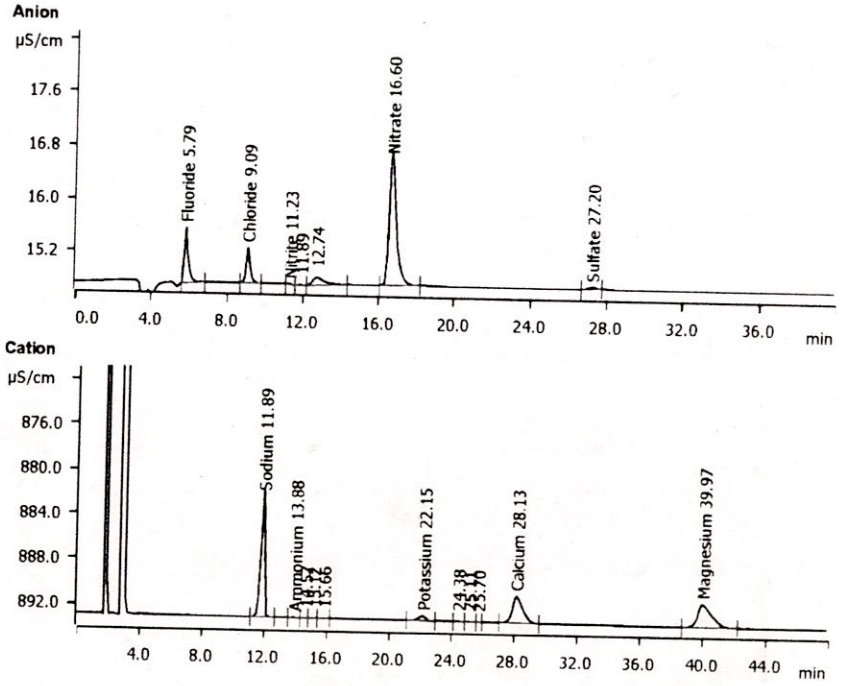

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N-25

Anion

Component name	Retention time min	Height µS/cm	Area (µS/cm) x min	Concentration mg/L
Fluoride	5.79	0.273	0.054	0.286
Chloride	9.08	2.061	0.469	3.827
Nitrite	11.01	0.006	0.001	0.017
Nitrate	16.73	0.011	0.004	0.055
Sulfate	27.72	0.012	0.012	0.137

Cation

Component name	Retention time min	Height µS/cm	Area (µS/cm) x min	Concentration mg/L
Sodium	11.94	7.470	2.120	18.925
Ammonium	13.95	0.733	0.227	4.570
Potassium	22.31	0.161	0.090	1.690
Calcium	28.45	2.370	1.823	22.822
Magnesium	40.83	1.521	1.494	8.426

% Error--------------------------------------.1.571
Sample pH---------------------------------------7.2
M-alkalinity as CaCO3----------------------------136.657
P-alkalinity as CaCO3----------------------------0.000
Total Hardness by IC----------------------------91.611

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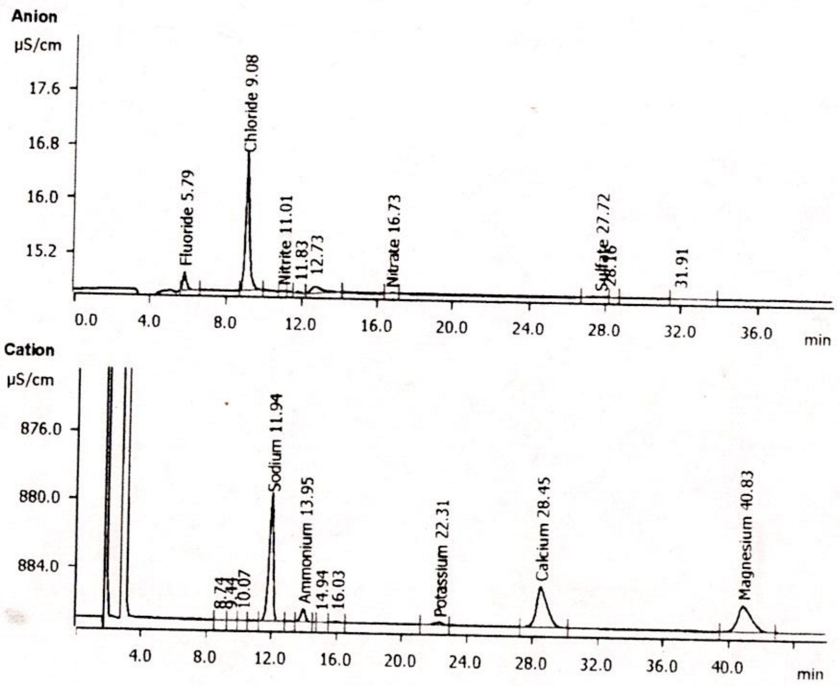

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N-26

Anion

Component name	Retention time min	Height µS/cm	Area (µS/cm) x min	Concentration mg/L
Fluoride	5.80	0.237	0.047	0.369
Chloride	9.07	0.423	0.097	1.188
Nitrite	11.18	0.014	0.006	0.124
Nitrate	16.59	0.407	0.182	3.940
Sulfate	27.77	0.008	0.006	0.095

Cation

Component name	Retention time min	Height µS/cm	Area (µS/cm) x min	Concentration mg/L
Sodium	11.90	4.231	1.176	15.743
Ammonium	13.95	0.029	0.009	0.259
Potassium	22.23	0.077	0.041	1.154
Calcium	28.54	2.145	1.616	30.339
Magnesium	40.76	1.627	1.607	13.595

% Error--.1.610

Sample pH---------------------------------------7.3

M-alkalinity as CaCO3----------------------------156.614

P-alkalinity as CaCO3----------------------------0.000

Total Hardness by IC-----------------------------131.634

NIH IC REPORT

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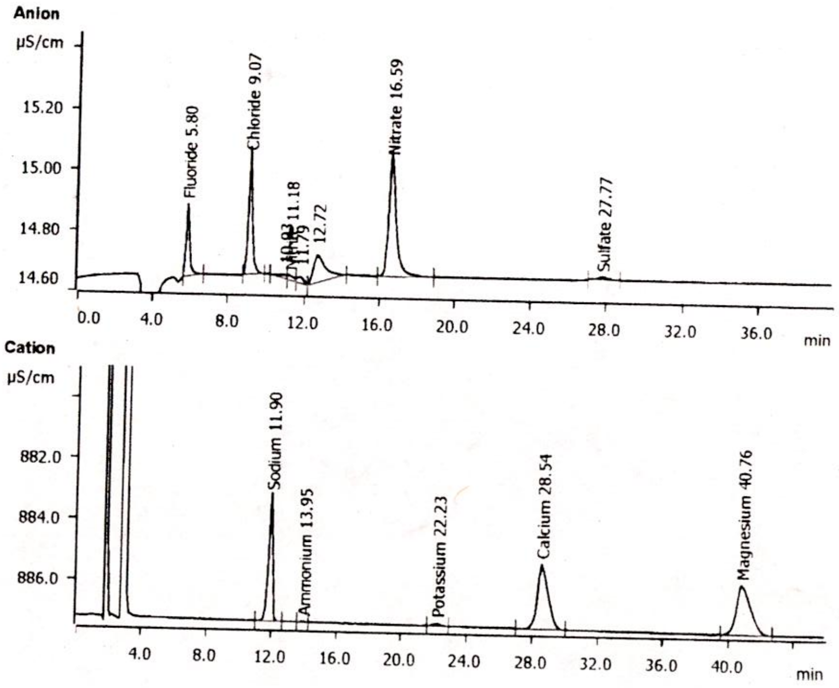

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DISCUSSION

The purpose of the present study was to characterized the ground water samples of Guwahati.

Six sample named (N-21, N-22, N-23, N-24, N-25, N-26) having pH =8.2, 7.1 , ,7.6, 7.2, 7.2,7.3 and ALKALINITY = 135.200, 173.343, 154.457, 80.271, 136.657, 156.614 and HARDNESS = 68.587, 157.826, , 144.897, 53.312, 91.611, 131.634 respectively were analyzed as per IS specifications. The anions and cations were determined by ion chromatography, specifically by titrimetric method.

We observed from the result that the hydrogen ion concentration in the all collation water sample were nor too high and neither too low. The concentrations obtained were with in the permissible limits that is approximately 6.5-8.5 pH (for drinking water).

While other parameter with in the range as per IS specification, although a few anions and cations were formed to be present in the high concentration in the water sample.

Hydro-chemistry of Ground water samples of Guwahati were studied. The nature of pH, HARDNESS, variable ALKALINITY and concentration of cation and anions were observed typically of Ground water samples.

CONCLUSION

ysis of six water samples of Guwahati were performed at National Institute ydrology, Roorkee according to standard method of IS for their pH, alinity, hardness, major cations and major anions. The results of all the ameters are found within the permissible limit of BIS .Thus all the six und water samples can be recommended for potable purpose.

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