**BASIC PHYSICOCHEMICAL ANALYSIS OF WATER SAMPLES COLLECTED FROM EAST SIKKIM****Rishi Choudhury, Koushal Singh, Utsav Sharma, Puja Newar, Arzoo Newar and Dipankar Nath\***

Department of Pharmaceutical Analysis and Quality Assurance, Himalayan Pharmacy Institute, Majhitar, East Sikkim, India.

**\*Corresponding Author: Dipankar Nath**

Department of Pharmaceutical Analysis and Quality Assurance, Himalayan Pharmacy Institute, Majhitar, East Sikkim, India.

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**ABSTRACT**

Water is one of the most important substance found in nature. It is present almost everywhere. All animals, plants and humans need water to survive. Water has been important for us for thousands of years. Without water there would be no life on earth. Chemically water is composed of hydrogen and oxygen. It exists in gaseous, liquid, and solid states. Water is one of the major solvent used up the pharmaceuticals. Water plays an important role in the pharmaceutical industry. It may be used as an excipient, or used for reconstitution of products, during synthesis, during production of finished product, or as a cleaning agent for rinsing vessels, equipment and primary packing materials. Depending on the quality of water purification steps are decided. Our study was aimed to analyze the basic physicochemical chemical Parameters (qualitative as well as quantitative) of water samples collected from East Sikkim, India. As Sikkim is occupied by number Chemical and pharmaceutical companies. So our aim was to ensure about the safety aspects of water collected from different source by comparing them samples with Packaged drinking water.

**KEYWORDS:** Water analysis, Physicochemical Parameters, Limit test, Qualitative and Quantitative analysis.**INTRODUCTION**

Water is one of the most important substances we know. It is present almost everywhere. All animals, plants and humans need water to survive. Water has been important for us for thousands of years. Without water there would be no life on earth. Chemically water is composed of hydrogen and oxygen. It exists in gaseous, liquid, and solid states. It is one of the most plentiful and essential of compounds. A tasteless and odorless liquid at room temperature, it has the important ability to dissolve many other substances. Indeed, the versatility of water as a solvent is essential to living organisms. Life is believed to have originated in the aqueous solutions of the world's oceans, and living organisms depend on aqueous solutions, such as blood and digestive juices, for biological processes.<sup>[1]</sup>

Water plays an important role in the pharmaceutical industry. It may be used as an excipient, or used for reconstitution of products, during synthesis, during production of finished product, or as a cleaning agent for rinsing vessels, equipment and primary packing materials etc. There are many different grades of water used for pharmaceutical purposes. Several are described in USP monographs that specify uses, acceptable methods of preparation, and quality attributes. These water can be

divided into two general types: bulk waters, which are typically produced on site where they are used; and packaged waters, which are produced, packaged, and sterilized to preserve microbial quality throughout their packaged shelf life. There are several specialized types of packaged waters, differing in their designated applications, packaging limitations, and other quality attributes. Different grades of water quality are required depending on the different pharmaceutical uses. Control of quality of water, in particular, the microbiological quality, is a major concern and the pharmaceutical industry devotes considerable resource to the development and maintenance of water purification systems.<sup>[2]</sup>

As we have seen that water plays an important role in pharmacy, it becomes very important to ensure that water which is used is of optimum quality. We know that due to rapid industrialization and exploding population the demand for water supply has increased enormously. Hence, the availability of fresh water has become a major cause of concern due to increased pollution of rivers by industrial and domestic discharge. This discharge of effluents results in the increase or decrease of various physicochemical parameters, which makes it unfit for human consumption.

Sikkim is well known throughout for its rich fauna and flora. About 70% of its total area is filled with forests. Sikkim is also known for its rich source of medicinal plants hence making it a major site of attraction for various researchers and scientists. The environmental and climatic conditions of the state make it best suitable for industrialization, hence attracting a lot of pharma giants. The increase in the number of industries has led to the increase in dumping of wastes in the streams or rivers which have affected the quality of water to some extent. The eastern region of Sikkim has a large number of industries located in the area. Hence it is important to analyze the water quality and keep a check on it.

Various physical, chemical, biological and microbiological parameters, which are essential to be checked before it is fit for human consumption as well as for the manufacturing of drugs, as listed as follows:

#### Physical Parameters

1. Temperature
2. Colour
3. Odour
4. Taste
5. Turbidity
6. Transparency
7. Conductivity
8. Total solid
9. Total dissolved solids
10. Total suspended solids
11. Velocity

#### Chemical Parameters

1. pH
2. Dissolved Oxygen (DO)
3. Biochemical Oxygen Demand (BOD)
4. Chemical Oxygen Demand (COD)
5. Carbon dioxide
6. Alkalinity
7. Hardness
8. Various metals and nonmetals like calcium, magnesium, chlorides, sulfates, nitrite, phosphorus, sodium, potassium and nitrates
9. Tannin and lignin
10. Oil and grease
11. Heavy metals like chromium, iron, arsenic, lead, mercury, manganese, cadmium and zinc.

#### Biological and microbiological analysis

1. Plankton
2. Nekton
3. Neuston
4. Benthos.<sup>[3]</sup>

Phadatare *et al.* (2016) stated that water quality is directly related to the physical, chemical, biological and radiological property of water. These properties of water are affected because of the pollution of water due to various human activities. She studied how disposal of pollutant in the water bodies changes the standard

quantity of parameters in water. There are various parameters which can be assess for measurement of quality of water but when consideration of all parameters may generate complexity towards quality. So, development of Water Quality Index (WQI) is the quite popular method in water quality assessment.<sup>[4]</sup> Singh *et al.* (2015) stated how water quality analysis is one of the most important aspects in surface water studies. water quality is a critical factor for assessing the pollution level. In his study the water samples were collected from 12 different sampling stations for evaluate the water quality status of river Narmada during September 2010. A total 16 water quality minimum and maximum value of air temperature, water temperature, turbidity, pH, electrical conductivity, total dissolved solids, free carbon dioxide, total alkalinity, chloride, total hardne Calcium Hardness dissolved oxygen, nitrate, orthophosphate, biochemical oxygen demand, chemical oxygen demand were noted.<sup>[5]</sup> Rahmanian *et al.* (2015), The drinking water quality was investigated by him in suspected parts of Perak state, Malaysia, to ensure the continuous supply of clean and safe drinking water for the public health protection. In this regard, a detailed physical and chemical analysis of drinking water samples was carried out in different residential and commercial areas of the state.<sup>[6]</sup>

Our study was carried out to assess the basic Physico-chemical Parameters of waters collected from different sources of East Sikkim which is a remote village in the northeastern part of India. More over this particular area is surrounded with good number of pharmaceutical industry too. So our study can help to get some data regarding the quality of water in the surroundings areas which is used up the local people on their day to day life. So we have collected water from two different sources in and around East Sikkim and chosen for our study.

#### MATERIALS AND METHODS

##### Sample collection and preservation

Water samples were collected directly from Khanikhola and Teesta and temperatures were recorded. The samples were preserved at Room temperature (approximately 38°C) in plastic bottles.

##### Physicochemical parameters

###### Colour

Water which was collected was found to be turbid because of the presence of various contaminants and microorganisms. The color of the water was checked by Visual method. In this method Specified quantity of water sample was first centrifuged then transferred in a Nessler's Cylinder and the color was then compared in bright sunlight.<sup>[3]</sup>

###### Odour

As the sensitivity of analyzing the odor varies from person to person, hence this testing is totally based on arbitrary comparisons as more absolute unit or base for odor exist. This method involves diluting in a particular

ratio of sample with odor free water until the least perceptible odor can be determined (approx. 100 ml). then different dilution samples are heated (approx. 60°C) over hot plate. The observations are then made from the lower concentration samples to higher concentration samples. As the sensitivity of odor varies from person to person, it is advisable to make observation from at least 5-6 persons.<sup>[3]</sup>

### Temperature

The water samples were collected in a plastic bottle directly from the source and the temperature was measured instantly with the help of a thermometer.<sup>[3]</sup>

### Determination of conductivity

**Instrument used:** Digital Conductivity meter (Labtech Instruments).

Conductivity refers to the degree at which a Specific material conducts electricity. The conductivity of the samples was checked by using Conductivity meter.

### Procedure

- Calibration of Conductivity meter: Calibration of conductivity meter was done by using 0.01 M KCl (0.5232gm in 1L of distilled water which is equal to 1000µS/cm).
- Conductivity Sensor was plugged into the data collection system, and turn on the system. The Conductivity Sensor does need to be calibrated for general studies. Pressed the button for µS/cm range for sample. If it was found that the Conductivity Sensor factory calibration is inadequate, calibration of conductivity Sensor was done using as standard value.
- Display was set to show conductivity measurement.
- Immersed the end of Conductivity or Salinity probe into the water to be measured. When the reading stabilizes, conductivity reading was recorded.<sup>[7,8,9]</sup>

**Note:** It's important to immerse the wholes near the end of the sensor. Probes need to be Swirled to ensure any air bubbles are removed from the end of the probe.

### Determination of pH

**Instrument used:** Digital pH meter (Thermo Instruments) pH is a scale of acidity from 0 to 14. It tells how acidic or alkaline a substance is. More acidic solutions have lower pH. More alkaline solutions have higher pH. The pH of the water samples was determined by Using Digital pH meter.

**Calibration of Digital pH meter:** pH meter was calibrated by using buffer solution of pH 4,7 and 9.2. Temperature was adjusted and was maintained at 25°C.

### Procedure

- Water Samples were stirred vigorously using a clean stirring rod.

- Pour about 40ml ± 5ml sample into the glass beaker and the beaker was covered by using watch glass.
- Let the sample stand for a minimum of one hour to allow the temperature to stabilize, stirred it occasionally while waiting. Measured the temperature of the sample and adjusted the temperature controller of the pH meter to that of the sample temperature.
- This adjustment should be done just prior to testing on meters with an automatic temperature control, followed the manufacturer's instructions.
- Immerse the electrode(s) of the p<sup>H</sup> meter into the water sample and turn the beaker slightly to obtain good contact between the water and the electrode(s).
- The electrode(s) require immersion 30 seconds or longer in the sample before reading to allow the meter to stabilize. If the meter has an auto read systems, it will automatically signal when stabilized.
- Read and recorded the pH value to the nearest tenth of a whole number. If the pH meter reads to the hundredth place, a round off rule will apply as follows If it is greater than 5, round the tenth place digit up one unit. If the hundredth place digit equals 5, round the tenth place digit to the nearest even number.
- Electrode was rinsed well with distilled water, dab lightly with tissues to remove any film formed on the electrode(s).<sup>[10]</sup>

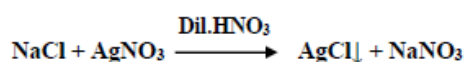
### Limit Tests

Limit tests are quantitative or semi quantitative tests to identify and control small quantities of impurity which are likely to be present in the substance. The quantity of impurities in a substance is often small but can cause errors in different circumstances. To avoid these errors, specific limit tests are performed. We performed a few of them which are listed below, Limit test of Chloride, Sulphate, Iron and Heavy metals was performed by using Nessler Cylinder whereas Limit test of Arsenic was performed by using Gutzeit Test.<sup>[11]</sup>

### Limit test for chloride

#### Principle

The limit test for chloride has been based upon the simple reaction between silver nitrate and soluble chlorides to obtain silver chloride which is insoluble in dilute nitric acid which precipitates and appear as white slid particles (opalescence). The silver chloride produced in the presence of dilute nitric acid makes the test solution turbid, the extent of turbidity depending upon the amount of chloride present in the substance is compared with a standard opalescence produced by the addition of silver nitrate to a standard solution having a known amount of chloride and the same volume of dilute nitric acid as used in the test solution. If the turbidity from the samples has been less than the standard turbidity, the sample will pass the limit test and vice versa.

**Reaction involved****Chemical Requirements**

Sodium chloride  
Nitric acid(dilute)  
Silver nitrate

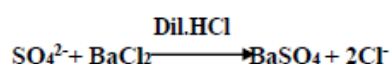
**Procedure****Table 1: Procedure for limit test for Chloride.**

| Standard   | Test   |
|--|--|
| 1 ml of 0.05845% w/v solution of NaCl was taken in a Nessler's cylinder and 10 ml of HNO <sub>3</sub> was added.   | Specified Volume of water sample was taken in a Nessler's cylinder and 10 ml of HNO <sub>3</sub> was added.  |
| 1 ml of 0.1 M AgNO <sub>3</sub> solution was added to the above solution and stirred properly and the volume was made up to 50 ml with distilled water, it was allowed to stand for 5 minutes. Turbidity/ opalescence was observed | 1 ml of 0.1 M AgNO <sub>3</sub> solution was added to the above solution and stirred properly and the volume was made up to 50 ml with distilled water, it was allowed to stand for 5 minutes. Turbidity/ opalescence was observed |
| The turbidity Produced by the Standard and Samples were compared. <sup>[12]</sup>  |  |

**Limit test for Sulphate****Principle**

The limit test for sulphate is carried out on the basis of the reaction between barium chloride and soluble sulphates in presence of dilute hydrochloric acid. Then, the comparison of the turbidity produced by a given amount of the substance is done with a standard turbidity obtained from a known amount of sulphate and same volume of dilute hydrochloric acid have been added to both the solutions. The barium chloride test solution in the I.P. has been replaced by Barium Sulphate Reagent which is having barium chloride, Sulphate-free alcohol and a solution of potassium sulphate. Potassium sulphate has been added to increase the sensitivity of the test. The ionic concentrations in the reagent has been so adjusted that the solubility of barium sulphate gets exceeded, and the very small amount of barium sulphate present in the reagent acts as a seeding agent for the precipitation of barium sulphate, if sulphate is present in the substance under test. Alcohol helps to prevent super-saturation and thus produces a more uniform opalescence.

The substance passes the limit test if it produces a turbidity that is less than the standard.

**Reaction involved****Chemical Requirements**

- Barium Chloride
- Dilute hydrochloric acid
- Sulphate-free alcohol
- Potassium sulphate.

**Procedure**

**Preparation of BaSO<sub>4</sub> Reagent:** 15ml of 0.5M BaCl<sub>2</sub>, 55ml of distill water and 20ml sulphate free alcohol was mixed and to this 5ml 0.0181% w/v K<sub>2</sub>SO<sub>4</sub> as added. The volume was made up to 100ml with distilled water and mixed.

**Table 2: Procedure for limit test for Sulphates.**

| Standard   | Test  |
|--|---|
| In a 50 ml Nessler's cylinder, a mixture of 1 ml of 0.1089% w/v solution of K <sub>2</sub> SO <sub>4</sub> + 2 ml HCl + 5 ml BaSO <sub>4</sub> reagent was added and made up the mark with distilled H <sub>2</sub> O. | In a 50 ml Nessler's cylinder a mixture of specified quantity of water sample + 2 ml HCl + 5 ml BaSO <sub>4</sub> reagent was added and made up the mark with distilled H <sub>2</sub> O. |
| Resulting solution was allowed to stand for 5 minutes. Turbidity/ opalescence was observed   | Resulting solution was allowed to stand for 5 minutes. Turbidity/ opalescence was observed  |
| The turbidity Produced by the standard and the samples were compared. <sup>[12]</sup>  |   |

**Limit test for Iron****Principle**

The limit test for iron is based on the reaction of iron in an ammonical solution in presence of citric acid, with thioglycollic acid when a pale pink to deep reddish purple colour is formed due to ferrous compounds. The color produced from a special amount of substance from the test is compared by viewing vertically with a standard (ferric ammonium sulphate). If the color from test solution is less dark than the standard, then the sample passes the limit test. Citric acid does not allow

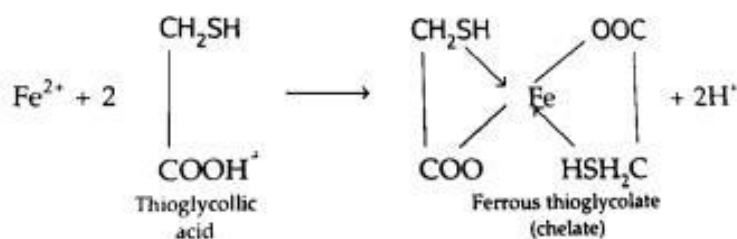
the precipitation of iron by ammonia by forming a complex with it. The color due to ferrous compound gets destroyed by oxidizing agents and alkalis. The state of oxidation of iron has been immaterial as Iron (III) gets reduced to Iron (II) by thioglycollic acid.

Thioglycollic acid, (HS)CH<sub>2</sub>COOH, is a useful analogue of glycolic acid, CH<sub>2</sub>(OH)COOH, which is prepared by the action of potassium hydrogen sulphide on monochloroacetic acid.

It is a colorless liquid having an unpleasant odor. The thioglycollic acid test for iron has now largely replaced former test which was based on the formation of the highly colored ferric thiocyanate by reaction between ammonium thiocyanate and iron(III) salts. It is believed that the thioglycollic acid test is more sensitive than the ammonium thiocyanate test.

The color produced from a specified amount of the substance is compared with the standard color obtained from a known amount of iron under exactly similar conditions. This test is very sensitive and interference with any other cations is eliminated by making use of 20% citric acid solution, which forms a complex with other metal cations.

#### Reactions involved



#### Chemical requirements

- Ferric ammonium sulphate
- Citric acid (Iron-free)
- Thioglycollic acid
- Ammonia

#### Procedure

**Preparation of standard solution of Iron:** It was prepared by adding 0.273 g of ferric ammonium sulphate  $[\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]$  to 1.5 ml of HCl and adding sufficient water to produce 1000 ml. Each ml of solution contains 0.02 mg of iron.

**Table 3: Procedure for limit test for iron.**

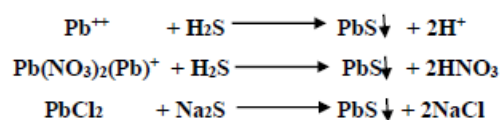
| Standard  | Test  |
|---|---|
| In a 50 ml Nessler's cylinder, a mixture of 2 ml of standard solution of iron + 20 ml water + 2 ml of 20% w/v (iron-free) citric acid + 2 drops of thioglycollic acid; solution made alkaline with ammonia and adjust the volume to 50 ml with distilled water. | In a 50 ml Nessler's cylinder a mixture of Specified quantity water sample+ 20 ml water + 2 ml of 20% w/v (iron-free) citric acid + 2 drops of thioglycollic acid; solution made alkaline with ammonia and adjust the volume to 50 ml with distilled water. |
| Resulting solution was allowed to stand for 5 minutes. Pinkish-purple color was developed.  | Resulting solution was allowed to stand for 5 minutes. Pinkish-purple color was developed.  |
| The samples and standard were viewed vertically and the intensity of the colors were compared. <sup>[11]</sup>  |   |

#### Limit test for heavy metals

##### Principle

The limit test for heavy metals is designed to determine the content of metallic impurities that are colored by hydrogen sulphide or sodium sulphide under the condition of the test. The heavy metals (metallic impurities) may be iron, copper, lead, nickel, cobalt, bismuth, antimony, etc. The method is based upon the reaction of the heavy metal ion with hydrogen sulphide leading to the formation of heavy metal sulphides. The metal sulphides remain distributed in a colloidal state and give rise, to a brownish coloration. The color produced in the test solution is compared to that of standard solution containing a definite amount of lead.

#### Reactions involved



- Lead nitrate
- Nitric acid
- Acetic acid
- Ammonia solution
- Dilute hydrochloric acid
- Ferrous sulphide

#### Procedure

**Preparation of Standard Lead solution:** In a 250ml of volumetric flask, 0.4 gm of lead nitrate was taken in distilled water and a 2 ml of nitric acid was added on to it and finally volume was adjusted up to the mark with distilled water.

**Preparation of Hydrogen Sulphide solution:** Weigh Approximately 1 gm of ferrous sulphide and transferred in a beaker containing 20 ml of dilute HCl.



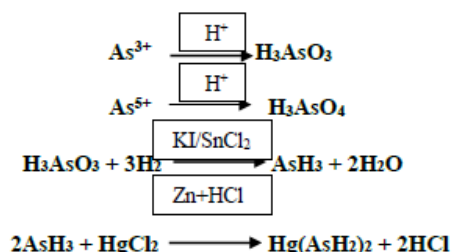
**Table 4: Procedure for limit test for heavy metal.**

| Standard   | Test  |
|--|---|
| In a 50 ml Nessler's cylinder, a mixture of 2 ml of standard lead solution+25 ml Distilled water was added. The pH was adjusted between 3 - 4 by adding acetic acid or ammonia solution. | In a 50 ml Nessler's cylinder, a mixture of specified amount of water sample+25 ml Distilled water was added. The pH was adjusted between 3 - 4 by adding acetic acid or ammonia solution |
| To the above solution, 10 ml of freshly prepared hydrogen sulphide solution was added. Mixed well and made up the volume to 50 ml with distilled water.                                  | To the above solution, 10 ml of freshly prepared hydrogen sulphide solution was added. Mixed well and made up the volume tom 50 ml with distilled water.                                  |
| The intensity of coloration of standard and samples were compared. <sup>[11]</sup>   |   |

**Limit test for arsenic****Principle**

Arsenic is a well-known undesirable and harmful impurity which is present in most substances. Many qualitative and quantitative tests for arsenic are known. However, the pharmacopoeias method is based on the Gutzeit test, using the Gutzeit apparatus.

The principle is based on Gutzeit test wherein, all arsenic present is duly converted into arsine gas (AsH<sub>3</sub>) by subjecting it to reduction with zinc and hydrochloric acid. The arsenious present gets converted to arsine gas easily with the help of reducing agents. For arsenic present in the sample, it reacts with hydrochloric acid to give arsenic acid, which gets reduced, by reducing agents, to arsine gas. When this gas is passed over mercuric chloride paper, it produces a stain, which ranges in colour from yellow to brown, the intensity and length of which are proportional to the amount of arsenic present. B.P. suggests the use of mercuric chloride paper instead of mercuric bromide paper. The Gutzeit apparatus is prepared for standard and the samples. A standard stain prepared from a definite quantity of arsenic, is used for comparison. The stain produced by the test is compared to the stain produces by the standard. If the stain produced by the sample is lesser than that of the standard, then the sample passes the limit test.

**Reactions involved****Chemical requirements**

- Arsenic trioxide
- Stannous chloride
- Potassium iodide
- Zinc
- Mercuric chloride
- Lead acetate.

**Procedure**

**Preparation of Stannated Hydrochloric Acid:** It was prepared from solution of stannous chloride by adding an equal volume of hydrochloric acid, boiling down to the original volume and filter through a fine-grained filter paper.

**Standard Arsenic Solution:** 0.33gm of AsO<sub>3</sub>(arsenic trioxide) was dissolved in 5ml of 2M NaOH and dilute to 250ml with distilled water. 1ml of above solution was then taken in a 100ml volumetric flask with distilled water.

**Table 5: Procedure for limit test for arsenic.**

| Standard   | Test   |
|--|--|
| A known amount dilute arsenic standard solution was kept in the wide open glass bottle of the apparatus.   | Specified amount of water sample was kept in wide open glass bottle of the apparatus.  |
| To this, 10 ml of stannated HCl, 5ml of 1M KI and 10gm of granulated zinc was added.   | To this, 10 ml of stannated HCl, 5ml of 1M KI and 10gm of granulated zinc was added.   |
| Above solution was kept aside for 40 minutes and then the mercuric chloride paper was removed slowly from between the bungs to see the intensity of the stain. | Above solution was kept aside for 40 minutes and then the mercuric chloride paper was removed slowly from between the bungs to see the intensity of the stain. |
| The intensity of the stain Produced by the standard was compared with the test samples. <sup>[11]</sup>  |  |

**Determination of acidity of water****Principle**

Acidity in water sample is due to the presence of carbon dioxide, mineral acid and hydrolyzed salts. Acidity in water not only makes it unsuitable for the growth of

aquatic plants fish and other useful microorganisms but also for domestic consumption and irrigation.

Acidity in water was determined by titrating it against standard alkali solution using methyl orange as an

indicator (pH 4.5). Acidity thus determined is called the methyl orange acidity. Phenolphthalein acidity also called total acidity is determined by titrating the water samples against standard alkali using phenolphthalein as indicator (pH range 8.3).

#### Chemical requirements

Standard N/20 Sodium hydroxide solution.  
Phenolphthalein indicator.

#### Methyl orange indicator

##### Procedure

- In a 250 ml conical flask 100ml of water sample was taken, 1-2 drops of N/10  $\text{Na}_2\text{S}_2\text{O}_3$  was added to destroy any residual chlorine.
- 1-2 drops of methyl orange indicator to it, If the solution turns yellow then methyl orange acidity is absent.
- But if the solution turns pink then it was titrated against N/20 Standard NaOH solution until the pink colored solution changes to yellow. Noted the volume of NaOH solution used.
- Now few drops of phenolphthalein indicator were added to the yellow colored solution and then titrate with N/20 NaOH solution until the pink color produced persists for few seconds. Noted the volume of NaOH used in the titration.<sup>[13]</sup>

#### Determination of permanent hardness

##### Principle

This determination is based on the precipitation of calcium and magnesium salts in the form of carbonates by the action of excess standard  $\text{Na}_2\text{CO}_3$  solution. After the separation of the precipitate the residual unchanged  $\text{Na}_2\text{CO}_3$  is titrated with HCl solution in the presence of methyl orange and the volume of  $\text{Na}_2\text{CO}_3$  solution taken for precipitation of the calcium and magnesium salts is found by difference. The hardness is easily calculated from the result. After addition of  $\text{Na}_2\text{CO}_3$  the solution is evaporated to dryness, when the acid salts  $\text{Ca}(\text{HCO}_3)_2$  and  $\text{Mg}(\text{HCO}_3)_2$  decompose completely to form insoluble carbonates. Therefore, this method gives not the total but only the permanent hardness of water.

#### Chemical requirements

Sodium Carbonate Hydrochloric acid Methyl orange.

##### Procedure

- Pipetted out 100ml of the water into a porcelain dish and from the burette an exactly measured volume (10-25 ml) of approximately 0.1N  $\text{Na}_2\text{CO}_3$  solution was added.
- Then evaporate the mixture until it dries off completely.
- The dry residue was then treated with 15-20 ml of distilled water free from carbon dioxide to extract residual (excess)  $\text{Na}_2\text{CO}_3$ .
- Filter off the undissolved residue consisting of calcium and magnesium carbonate and wash the residue three to four times with the same water,

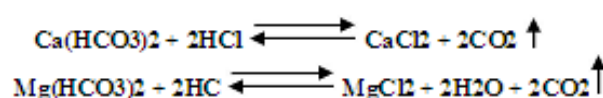
collected the filtrate and washings in a conical flask for titration.

- At the end of washing, 1-2 drops of methyl orange is added to the solution and titrate it with hydrochloric acid.
- Now determined the relationship between the equivalent volumes of  $\text{Na}_2\text{CO}_3$  and HCl solutions. The simplest way to do this is to measure out with a burette into a flask the same volume of  $\text{Na}_2\text{CO}_3$  solution as was used for the determination, and to titrate it (in duplicate or triplicate) with the HCl solution in the presence of 1-2 drops of methyl orange indicator.<sup>[14]</sup>

#### Determination of carbonate hardness

##### Principle

A known volume of water is titrated with hydrochloric acid in presence of methyl orange, when the following reaction takes place:



#### Chemical requirements

- Methyl orange
- Hydrochloric acid

##### Procedure

Measured about 100-200 ml of the water in a Conical Flask, 2-3 drops of methyl orange Indicator were added, and titrated with standard 0.1N HCl solution.

Titration was repeated for two to three times and the average of the concordant results was taken.<sup>[14]</sup>

#### Estimation of chloride by Mohr's method

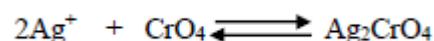
##### Principle

Chlorides are present in varying proportions in all natural waters. Chloride rich natural water through water soluble chloride salts from the surface of soil, through human excrete, through industrial waste and through artificial spray. Chlorides present in reasonable concentration (75-200mg/l) in water are not harmful for human consumption.

The determination of Chlorides involves the use of silver nitrate in the presence of potassium chromate as indicator. Initially white silver chloride is precipitated as indicated by its solubility product.



But when all the chloride ions have been precipitated a drop of silver nitrate in excess gives a red precipitate of silver chromate.



Therefore, it is necessary to perform blank titration. Volume of silver nitrate used in the blank titration is subtracted from the volume of silver nitrate used in titrating the sample.

#### Chemical requirements

- Silver Nitrate
- Potassium chromate
- Phenolphthalein
- Sodium carbonate

#### Preparation of Standard N/50 Silver Nitrate Solution:

Weigh accurately ( $169.9/50 \times 4 = 0.8495$ ) 0.85 gm of A.R Silver nitrate and transferred it in a 250 ml volumetric flask, distilled water was added Stopper the flask and shake thoroughly so that silver nitrate completely dissolves. Solution was made up to the mark by adding distilled water.

#### Preparation of Potassium chromate indicator:

Dissolved 5gm of  $K_2CrO_4$  in about 5ml of distilled water and few drops of silver nitrate solution was until a definite red ppt. forms. Let it stand for 12hrs and filter and finally dilute to 100ml with distilled water.

**Preparation of Phenolphthalein indicator:** Weigh 0.5gm of phenolphthalein powder and 50ml of Absolute ethanol was added, stir well to dissolve properly and made up the volume to 100ml with distilled water.

**Preparation of Standard N/50 Sodium carbonate Solution:** Weigh accurately ( $53/50 = 1.060$ ) 1.060gms of AR grade sodium carbonate and transfer it to a 1 L Volumetric flask. Few ml of distilled water was added, stopper the flask and shake thoroughly so that the alkali completely dissolves. Volume was made up to the mark by adding more distilled water.<sup>[15]</sup>

#### Procedure

- The given water sample was filtered so as to remove any suspended material. 50ml of this filtered sample was pipette out in a large porcelain dish and 3-4 drop of phenolphthalein indicator was added to it.
- A pink colour was develop, so dil.  $H_2SO_4$  was added drop wise until the pink colour just disappeared.
- If the pink colour is not obtained on adding phenolphthalein indicator than add 3-4 drop methyl orange indicator to the red colour solution obtained after adding methyl orange indicator add N/50  $Na_2CO_3$  solution until the colour of the solution changes to the orange. However, if the yellow colour solution is obtain on adding methyl orange than do not add  $Na_2CO_3$  solution.
- The resultant solution was transferred in a 250ml conical flask and 1ml of  $K_2CrO_4$  indicator was added to it.
- Slowly standard N/50  $AgNO_3$  solution was added from the burette with constant shaking. A white ppt. of silver chloride was obtained.

- The addition process was continued slowly till a red colour appeared in the flask, which disappeared on shaking. Then, silver nitrate solution was added drop by drop until a permanent reddish brown colour was obtained.
- The colour change may be more readily determine by performing the blank titration. The volume of silver nitrate used was noted and the process was repeated again till we got two concordant readings.

## RESULTS

### Physicochemical parameters

#### Color (visual method)

**Table 6: Colour Comparison by visual method.**

| Sample           | Inference   |
|------------------|-------------|
| Teesta Water     | Less turbid |
| Khanikhola Water | Turbid      |
| Drinking water   | Clear       |

#### Odour

**Table 7: Odour Comparison of collected Water Sample.**

| Sample           | Inference      |
|------------------|----------------|
| Teesta Water     | Characteristic |
| Khanikhola Water | Characteristic |
| Drinking water   | Characteristic |

**Table 8: Temperature of collected Water Sample.**

| Sample           | Inference |
|------------------|-----------|
| Teesta Water     | 19°C      |
| Khanikhola Water | 25°C      |
| Drinking water   | 27°C      |

The temperature of the samples were found to be of the order:

**Drinking water > Khanikhola Water > Teesta Water**

#### Determination of conductivity

**Table 9: Result for determination of conductivity.**

| Sample           | Conductivity( $\mu s/l$ ) |
|------------------|---------------------------|
| Teesta Water     | 699.4                     |
| Khanikhola Water | 598.2                     |
| Drinking water   | 530.1                     |

The Conductivity of the samples were found to be of the order:

**Drinking water > Teesta Water > Khanikhola Water**

#### Determination of pH

**Table 10: Result for determination of pH.**

| Sample           | pH    |
|------------------|-------|
| Teesta Water     | 6.554 |
| Khanikhola Water | 6.281 |
| Drinking water   | 6.143 |

The pH of the samples were found to be of the order:

**Teesta Water > Khanikhola Water > Drinking water**

#### Limit test for chloride



**Table 11: Result for limit test for Chloride.**

| Sample           | Inference              |
|------------------|------------------------|
| Standard         | Turbidity was observed |
| Teesta Water     | Turbidity was observed |
| Khanikhola Water | Turbidity was observed |
| Drinking water   | Turbidity was observed |

The turbidity of the samples were found to be of the order:

**Standard > Teesta Water > Khanikhola Water > Drinking water**

**Conclusion:** After Comparing the Turbidity of Samples with that of Standard, it was found that standard was more turbid as compare to the samples. Hence all the samples passes the limit test for Chloride.

#### Limit test for sulphate

**Table 12: Result for limit test of Sulfate.**

| Sample           | Inference              |
|------------------|------------------------|
| Standard         | Turbidity was observed |
| Teesta Water     | Turbidity was observed |
| Khanikhola Water | Turbidity was observed |
| Drinking water   | Turbidity was observed |

The turbidity of the samples were found to be of the order:

**Standard > Teesta Water > Khanikhola Water > Drinking water**

**Conclusion:** After Comparing the Turbidity of Samples with that of Standard, it was found that standard was more turbid as compare to the samples. Hence all the samples passes the limit test for Sulphate.

#### Limit test for iron

**Table 13: Result for limit test for iron.**

| Sample           | Inference                |
|------------------|--------------------------|
| Standard         | Pink colour was observed |
| Teesta Water     | Pink colour was observed |
| Khanikhola Water | Pink colour was observed |
| Drinking water   | Pink colour was observed |

The colour intensity of the samples were found to be of the order:

**Standard > Teesta Water > Khanikhola Water > Drinking water**

**Conclusion:** After Comparing the Intensity of the color of Samples with that of Standard, it was found that standard was more Colored as compare to the samples. Hence all the samples passes the limit test for Iron.

#### Limit test for heavy metals

**Table 14: Result for limit test for heavy metals(Pb).**

| Sample           | Inference                          |
|------------------|------------------------------------|
| Standard         | Greenish-black colour was observed |
| Teesta Water     | Greenish-black colour was observed |
| Khanikhola Water | Greenish-black colour was observed |
| Drinking water   | Greenish-black colour was observed |

The colour intensity of the samples were found to be of the order:

**Standard > Teesta Water > Khanikhola Water > Drinking water**

**Conclusion:** After Comparing the Intensity of the color of Samples with that of Standard, it was found that standard was more Colored as compare to the samples. Hence all the samples passes the limit test for Lead.

#### Limit test for arsenic

**Table 15: Result for limit test for arsenic.**

| Sample               | Inference             |
|----------------------|-----------------------|
| Standard             | Stain was observed    |
| Teesta Water         | Stain was observed    |
| Khanikhola Water     | Stain was observed    |
| Drinking water Water | No stain was observed |

The intensity of stain of the samples were found to be of the order:

**Standard > Teesta Water > Khanikhola Water > Drinking water**

**Conclusion:** After Comparing Colour Intensity of the stain Produced by the Samples with that of Standard on the mercuric chloride paper, it was found that standard produced more Intense stain as compare to the samples. Hence all the samples passes the limit test for Arsenic.

#### Determination of acidity of the sample

**Table 16: Result for determination of acidity of the different water samples.**

| Sample           | Acidity(ppm) |
|------------------|--------------|
| Teesta Water     | 2.75         |
| Khanikhola Water | 5.75         |
| Drinking Water   | 5.75         |

The order of acidity of the samples were found to be:

**Drinking water = Khanikhola Water > Teesta Water**

#### Determination of hardness of water sample

**Table 17: Result for estimation of carbonate hardness of different water sample.**

| Sample           | Carbonate hardness(mg-eq/litre) |
|------------------|---------------------------------|
| Teesta Water     | 5.18                            |
| Khanikhola Water | 6.28                            |
| Drinking Water   | 4.29                            |

The order of Carbonate hardness of the samples were found to be:

**Khanikhola Water > Teesta Water > Drinking water**

**Table 18: Result for estimation of permanent hardness of different water sample.**

| Sample           | Permanent hardness(mg-eq/litre) |
|------------------|---------------------------------|
| Teesta Water     | 2.56                            |
| Khanikhola Water | 1.89                            |
| Drinking Water   | 0.9                             |

The order of Permanent hardness of the samples were found to be:

**Teesta Water > Khanikhola Water > Drinking water**

**Estimation of chloride by mohr's method**

**Table 19: Result for Chloride estimation by Mohr's method.**

| Sample           | Chloride (ppm) |
|------------------|----------------|
| Teesta Water     | 78.1           |
| Khanikhola Water | 44.02          |
| Drinking Water   | 29.82          |

The order of Chloride content of the samples were found to be:

**Teesta Water > Khanikhola Water > Drinking water**

**Table 20: Summary of Experiments Performed.**

| Parametr                                     | Sample         |                   |                |
|--|----------------|-------------------|----------------|
|  | Teesta Water   | Khani khola water | Drinking water |
| Colour                                       | Less turbid    | Turbid            | Clear          |
| Odour  | Characteristic | Characteristic    | Characteristic |
| Temperature (oC)                             | 19o            | 25o               | 27o            |
| Conductivity( $\mu$ s/l)                     | 699.4          | 598.2             | 530.1          |
| pH   | 6.554          | 6.281             | 6.143          |
| Limit Test for Cl <sup>-</sup>               | pass           | pass              | pass           |
| Limit Test for SO <sub>4</sub> <sup>-</sup>  | Pass           | Pass              | Pass           |
| Limit Test for Iron                          | Pass           | Pass              | Pass           |
| Limit Test for Heavy Metals                  | Pass           | Pass              | Pass           |
| Limit Test for Arsenic                       | Pass           | Pass              | Pass           |
| Acidity(ppm)                                 | 2.75           | 5.75              | 5.75           |
| Carbonate Hardness(mg-eq/litre)              | 5.181          | 6.28              | 4.29           |
| Permanent Hardness(mg-eq/litre)              | 2.56           | 1.89              | 0.9            |
| Estimation of Chloride by Mohrs' method(ppm) | 78.1           | 44.2              | 29.82          |

## CONCLUSION

In Conclusion, we have successfully analyzed various Physicochemical Parameters of water samples Collected from two major water sources of East Sikkim.

Water Samples collected from Khanikhola and Teesta passes the limit tests for Chloride, Sulphate, Iron, Heavy metals and Arsenic. But it was observed that turbidity/Colour Intensity/ Stain produced by both the samples were more as compare to normal drinking water. So quantification of Sulphate, Iron, Heavy metals and Arsenic these can give a better idea regarding their concentration in water samples.

## SUMMARY

Water plays an important role in day to life, it becomes very important to ensure that water which is used is of optimum quality. We know that due to rapid industrialization and exploding population the demand for water supply has increased enormously. Hence, the availability of fresh water has become a major cause of concern due to increased pollution of rivers by industrial and domestic discharge. This discharge of effluents results in the increase or decrease of various physicochemical parameters, which makes it unfit for human consumption.

So our study mainly focused on the evaluation of quality parameters of water samples both qualitatively and quantitatively. Results of water analysis are summarized in the table.

P<sup>H</sup> Study confirmed us that all the water samples are in slight acidic range, can say near about neutral, whereas conductivity of Teesta water was higher as compare to the Khanikhola and Drinking water which gives us an indication that there may be presence of free ions and that was confirmed during Chloride estimation where we found Teesta water have higher concentration of Chloride as compare to Khanikhola and Drinking water.

Permanent hardness study confirmed us that Teesta water is Hard as compare to Khanikhola and Drinking water which was further supported by froth test.

So in future it will be better if we include some microbiological test in our experiment which can finally give us information regarding the usage of these water.

All the results are obtained in the lab condition.

We have done only qualitative analysis so our work can give a basis for the future regarding quantification of impurity and their limit assessment.

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