

STUDY MODULE- II

Water Chemistry

(Physico-chemical Analysis of Water)

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General Course Information

MODULE FOR ANALYSIS OF PHYSICO CHEMICAL PARAMETERS OF WATER

Coordinating Unit: Pakistan Council of Research in Water Resources

Duration: 6 months

Location: Islamabad

Units: 7

Course Description

Course Learning Outcomes: On successful completion of this course students will be able to:

- 1 Explain basic water chemistry
- 2 Identify the parameters that are involved in characterizing the drinking water safe or unsafe
- 3 Recognize the difference between physical and chemical parameters
- 4 Explain the importance of water quality tests and the use of different instruments for water quality tests in laboratory and on site testing.
- 5 Perform water quality testing in laboratory and on-site Learning resources

Learning and teaching activities: Students will be engaged in the diploma course by giving lectures, laboratory demonstration and field visits.

Assessment:

- Assessment must encourage and reinforce learning.
- Assessment must enable robust and fair judgments about student performance.
- Assessment practices must be fair and equitable to students and give them the opportunity to demonstrate what they have learned.
- Assessment must maintain academic standards.

Assessment Summary: Assessment will be purely done on the best analytical capabilities of student like working on the instrument, calibration, preparation of standards, reporting of result etc.

Student feedback: Feedback will be sought from students in a variety of ways including on-going engagement with staff, the use of group discussion and the use of Student Experience of Learning and Teaching.

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ABBREVIATIONS

Sr. No.	Abbreviation	Expanded Form
1	%	Percentage
2	µm	Micro meter
3	0C	Degree Celsius
4.	L	Liter
5.	Min	Minutes
6.	mL	Milliliter
7.	ppm	Parts per million
8.	gm	Gram
9.	mg	Milligram
10.	mg/L	Milligram per litre
11.	NGVS	No Guideline Value Set

Section A: Physical Parameters

1 Electrical Conductivity

1.1 Introduction:

Conductivity is a measure of the ability of water to pass an electrical current. (Figure 1) Because dissolved salts and other inorganic chemicals conduct electrical current, conductivity increases as salinity increases. Salts disassociate into positively and negatively charged ions in solution, and the ions conduct electricity. Inorganic dissolved solids such as chloride, nitrate, sulfate, and phosphate are present in water as negatively charged ions (anions). Sodium, magnesium, calcium, iron and aluminum are present in water as positively charged ions (cations). Pure water is a poor conductor of electricity.

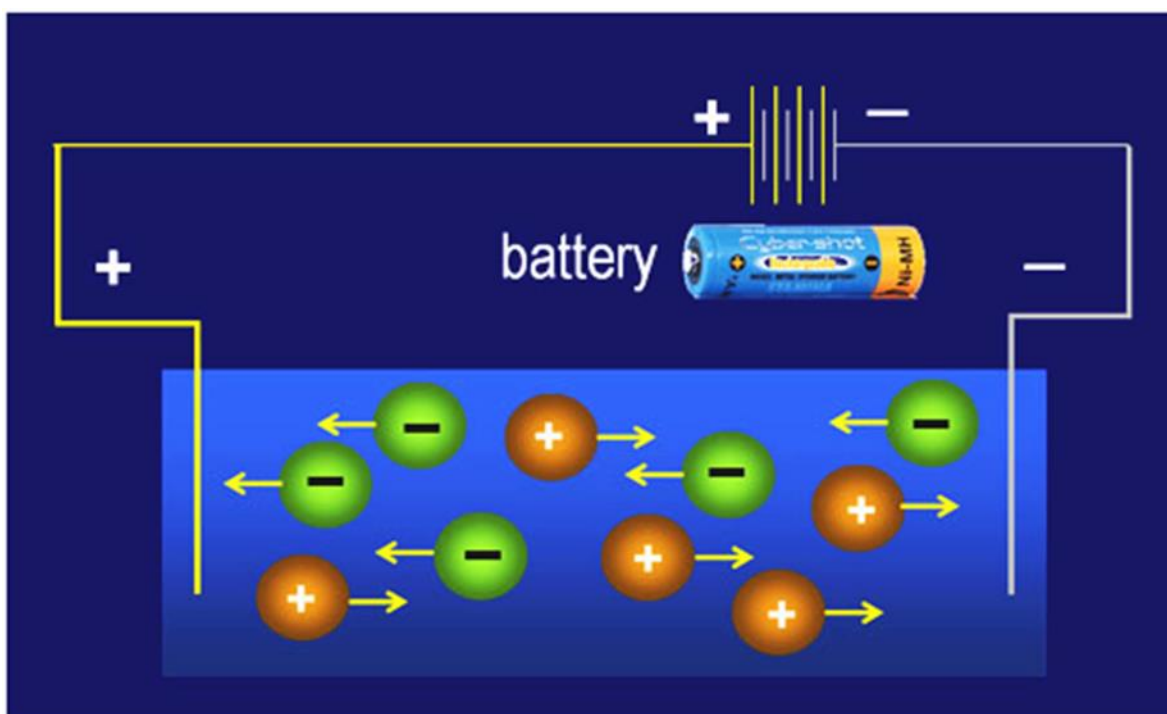


Figure 1: Flow of electric current

1.1.1 How does temperature affect electrical conductivity?

The higher the water temperature, the higher the electrical conductivity would be. The electrical conductivity of water increases by 2-3% for an increase of 1 degree Celsius of water temperature.

1.1.2 Why is it important to evaluate conductivity?

Conductivity is useful as a general measure of water quality. Significant changes in conductivity could then be an indicator that a discharge or some other source of pollution has entered the aquatic resource.

1.1.3 Permissible limits of Electrical Conductivity

- Natural drinking water (PSQCA/NSDWQ, 2010): NGVS
- Bottled water (PSQCA 2010): NGVS

1.2 Standard Operating procedure for analysis of Electrical conductivity

1.2.1 Testing Method

Electrometric method

1.2.2 Testing Instrument

EC Meter (JENWAY 4510) (Figure 2)



Figure 2: Electrical conductivity meter (JENWAY 4510)

1.2.3 Principle of Measurement

The Electrical Conductivity is a measure of ability of an aqueous solution to carry an electric current. This ability depends on presence of ions, mobility, and their total concentration, valence, and on the temperature of measurement. The more dissociated the electrolyte, the more specific conductance.

1.2.4 Preparation for EC Testing

1.2.5 Required Chemical and Reagents

- Potassium Chloride (anhydrous)
- Distilled water
- Preparation of electrical conductivity standard (1.413 mS/m or 1413 μ S/cm)

1.2.6 Equipment & Glassware

- Weighing Balance
- 1000 ml volumetric flask
- 1000 ml bottle container
- Temperature measuring Device

1.2.7 Preparation of Standards

- Weigh 745.6 mg (0.7456gm) KCl and dissolve in distilled water.
- Quantitatively transfer to 1000 ml volumetric flask.
- Dilute to mark with distilled water at 25 °C.
- Transfer to an appropriately labeled bottle

1.3 Calibration Procedure

- Turn “ON” the instrument.
- Select “SETUP” menu option for entering standard solution value.
- Select the Cal 1.
- Enter 1.413mS/m reading and move to exit.
- Moving the cursor to Cal menu option performs calibration.
- Immerse the conductivity cell into the prepared standard until end point symbol appears.
- Press the Enter key to calibrate and then move to exit option.

1.4 Testing

- Turn on the instrument by pressing -<SELECT MODE> and give it 5 minutes warm up time.
- Rinse the probe with Deionized water and check the calibration.
- Take 50 ml Deionized water in a beaker, immerse the probe in it and stir the blank until the instrument gives stable reading. (Maintain temperature at 25°C).
- Similarly take 50 ml sample in a beaker, rinse the probe with sample and dip the probe in it, continue stirring until the instrument gives stable reading of EC.

1.5 Expression of Results

The Electrical Conductivity is measured in $\mu\text{S}/\text{cm}$

1.5.1 Interpretation of Results

Compare the results with Water Quality Standards or Guidelines to predict the safe or unsafe level.

1.6 Checklist for testing of EC

Serial #	Tasks to be performed	Yes/ no
1	Introduction to the topic	
2	Instrument demonstration	
3	Reagents preparation	
4	Testing of water sample	
5	Quality control	
6	Reporting the results	

1.7 Planned Demonstration

Testing of EC in Chemical Lab (Reagents preparation, glassware washing, standards preparation, testing, quality control, calculations and reporting, units conversion)

1.8 Class Exercises

- surprised quiz
- handling of instrument
- analysis of unknown sample

1.9 Group project

Water Quality Assessment

2 TDS (Total Dissolved Solids)

2.1 Introduction

Total Dissolved Solids (TDS) are a measure of the amount of particulate solids (organic and inorganic materials, such as metals, minerals, and ions) that are in solution. This is an indicator of non-point source pollution problems associated with various land use practices. These materials, both natural and anthropogenic (made by humans), are mainly inorganic solids, with a minor amount of organic material. Depending on the type of water, TDS can vary greatly from a few milligrams per liter to percent levels (tens of thousands of milligrams per liter).

2.1.1 TDS source in water

TDS can come from just about anywhere, including minerals in springs from a water source, chemicals used to treat the water supply from sewage systems, runoff from road salts and yard chemicals or fertilizers, even the plumbing in your home.

Table 1: TDS Water Chart

TDS range	Effect on health
<50-250 ppm	considered low: missing in some of the minerals that are beneficial to your health
250-1000 ppm	Perfect: This level is the sweet spot for TDS in drinking water
1000-2000 ppm	Terrible: Not recommended or safe for house hold use
>2000 Worst:	A TDS level above 2000 ppm is completely unsafe and household filters cannot handle this level of contamination

2.1.2 Effects of TDS

- High levels of TDS affect the taste of drinking water.
- Hard water having high TDS collects in pipes and forms scale and buildup, which result in costly replacement for pipes and reduce the life of your appliances.
- Cooking with elevated TDS can change the taste of food.
- High TDS (greater than 1000 ppm) require further investigation for toxic particles such as heavy metals which are harmful for human health.

2.1.3 Ways to reduce TDS in water

A water treatment system is a great way to reduce or remove TDS from water. There are three TDS water filters in use which reduce high levels of total dissolved solids in tap and drinking water.

- Reverse osmosis (RO) system: This system forces water through a semipermeable membrane that eliminates total dissolved solids. It also uses a sediment and carbon filter to remove other forms of contaminants.
- Water Distiller: A water distiller removes contaminants through evaporation. As water evaporates, contaminants are unable to evaporate.
- Deionization (DI) system: Deionization systems remove total dissolved solids through ion exchange, using resins that control the electrical charge of ions. Water ions are used to replace charged ions from total dissolved solids. The water produced is highly pure, so DI cartridges are often referred to as high purity filters.

2.1.4 Permissible limits of TDS

- Natural drinking water (PSQCA/NSDWQ, 2010): 1000 ppm
- Bottled water (PSQCA 2010): 500 ppm

2.2 Standard operating procedure for analysis of TDS

Testing Method: Electrometric method

Testing Instrument: EC Meter (JENWAY 4510)

2.3 TDS measurement

The TDS measurement should be obtained with the conductivity meter by following these steps:

- Turn on the instrument by pressing -<SELECT MODE> and give it 5 minutes warm up time.
- Rinse the probe with Deionized water and check the calibration. After calibration select <TDS> mode.
- Take 50 ml Deionized water in a beaker, immerse the probe in it and stir the blank until the instrument gives stable reading. (Maintain temperature at 25°C).
- Similarly take 50 ml sample in a beaker, rinse the probe with sample and dip the probe in it, continue stirring until the instrument gives stable reading of TDS.

2.4 Quality Control

- Electrical Conductivity Meter is standardized by running known standard.
- Method blank is analyzed after every 10 samples
- Laboratory Control samples of 1413 $\mu\text{S}/\text{cm}$ analyzed with every batch of 10 samples.

2.5 Expression of Results

- TDS is measured in mg/l .

2.5.1 Interpretation of Results

Compare the results with Water Quality Standards or Guidelines to predict the safe or unsafe level.

2.6 Precautions

- Warm up instrument for 5 minutes before start working it.
- Rinse its probe properly with deionized water and select desired mode of working.
- The presence of particulate matter in the sample can lead to unstable and non-reproducible results. If necessary filter, or allow the particles to settle prior to immersion.

2.7 Checklist for testing of TDS

Serial #	Tasks to be performed	Yes/ no
1	Introduction to the topic	
2	Instrument demonstration	
3	Reagents preparation	
4	Testing of water sample	
5	Quality control	
6	Reporting the results	

2.8 Planned Demonstration

Testing of EC in Chemical Lab (Reagents preparation, glassware washing, standards preparation, testing, quality control, calculations and reporting, units conversion)

2.9 Class Exercises

- surprised quiz
- handling of instrument
- analysis of unknown sample

2.10 Group project

Water Quality Assessment

3 pH

3.1 Introduction

pH value is a good indicator of whether water is hard or soft. Danish chemist, S.P.L. Sorensen, was the first person to introduce the concept of pH scale. The U.S. National Bureau of Standards has defined pH values in terms of the electromotive force existing between certain standard electrodes in specified solutions. pH is a measure of hydrogen ion concentration, a measure of the acidity or alkalinity of a solution. The pH scale usually ranges from 0 to 14 (Figure 3). Aqueous solutions at 25°C with a pH less than 7 are acidic, while those with a pH greater than 7 are basic or

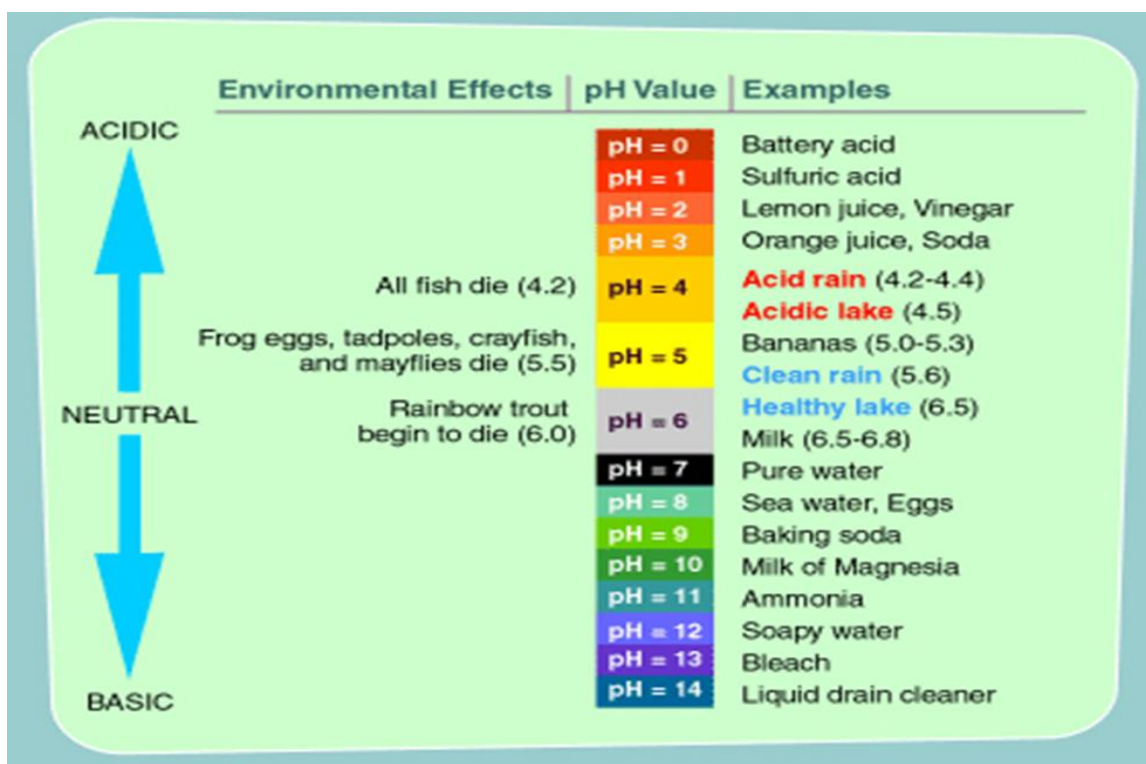


Figure 3: pH scale

alkaline. High-pH water has a slippery feel, tastes a bit like baking soda, and may leave deposits on fixtures. Low-pH water, on the other hand, may have a bitter or metallic taste, and may contribute to fixture corrosion.

3.1.1 Importance of pH

- In Agriculture: By determining the pH of the soil. We can find whether it is acidic or alkaline. This helps in deciding the type of fertilizer to be used and the types of crops to sown.
- In Biological process: By knowing pH we can adjust the medium of biological processes like fermentation, enzyme hydrolysis, sterilization etc.
- In corrosion research: By measuring the pH of sea-water, the effect of alkaline sea-water on the material used for building ships and submarines is studied.

3.1.2 Effect of pH on human body

The human body, its blood and its entire liquid media have a measurable pH. All cells, organs and bodily fluids need a specific pH to function at their best.

- When your body fluids contain too much acid, it's known as acidosis. Acidosis occurs when your kidneys and lungs can't keep your body's pH in balance.
- Alkalosis occurs when your body has too many bases. It can occur due to decreased blood levels of carbon dioxide, which is an acid. It can also occur due to increased blood levels of bicarbonate, which is a base

3.1.3 Permissible limits of pH

Natural drinking water (PSQCA/NSDWQ, 2010): 6.5-8.5

Bottled water (PSQCA 2010): 6.5-8.5

3.2 Standard Operating Procedure for Analysis of pH

3.2.1 Testing Method

Electrometric method

3.2.2 Principle of Measurement

The basic principle of electrometric pH measurement is the determination of activity of the hydrogen ions by potentiometric measurement using a standard hydrogen electrode and a reference electrode. Nowadays the glass electrodes are commonly used for the pH measurement. The electromotive force (e.m.f) produced in the glass electrode system varies linearly with pH.

3.2.3 Interference

- At pH value above 10, high sodium concentrations interfere with the measurement.
- Oil and grease may interfere by coating the pH electrode and causing a sluggish response.
- Temperature affects the pH values.

3.2.4 Testing Instrument

pH Meter (Figure 4)



Figure 4: pH meter

3.3 Standards and Reagents

Buffers of pH 4, 7 and 10 are prepared as following;

3.3.1 Preparation of Buffer pH 4

Weigh and dissolve 10.12g Potassium hydrogen phthalate in 1000 ml distilled water.

- Store buffer solutions in plastic bottles labeled with name of reagent & dates of preparation and expiry.

3.3.2 Preparation of Buffer pH 7

Weigh and dissolve 1.179g potassium dihydrogen phosphate and 4.303g disodium hydrogen phosphate in 1000 ml distilled water.

3.3.3 Preparation of Buffer pH 10

Weigh and dissolve 2.092g Sodium Bicarbonate and 2.640g sodium carbonate in 1000 ml distilled water.

3.4 Sample handling preservation

- Samples should be analyzed as soon as possible preferably in the field at the time of sampling.
- The sample containers should be filled completely and kept sealed prior to analysis.

3.5 Calibration Procedure of pH Meter

- Turn “ON” the pH meter by pressing power switch.
- Press “ON/OFF” button
- To calibrate the instrument, dip the electrode in pH 4, 7 or pH 10 buffers depending on the pH working range.
- Wait for the measured value to stabilize, and then adjust the screws to read the exact value of buffer at the measured temperature.
- The instrument is calibrated and ready for use.

3.6 Testing

- Turn on the instrument and give it 5 minutes warm up time. Press the desired mode of measurement.
- Rinse the electrode with deionized water and check the calibration by running known buffers of pH (4.00, 7.00 or 10.00).
- Take 50 ml sample in a beaker, immerse the probe and stir it until instrument gives stable pH at temperature of 25 °C.

3.7 Quality Control

- Method blank is analyzed after every 10 samples
- Laboratory Control samples of pH 7.00 and previously analyzed samples analyzed with every batch of 10 or less samples.

3.8 Expression of Results

There is no unit for measuring pH.

3.8.1 Interpretation of Results

Compare the results with Water Quality Standards or Guidelines to predict the safe or unsafe level.

3.9 Precautions

- Let the instruments warm up for five minutes before starting working on it.
- Rinse the probe properly before measurement.
- It should be calibrated with standards solution daily before use.
- If the probe has been left dry, condition it in pH (7) solution at least for one hour to reactivate it.
- If the measurements of different samples are taken successively rinse the probe tip thoroughly to eliminate cross contamination, after clearing rinse the probe tip with some of the sample to be measured.
- Before taking any measurement warm up the instrument for five minutes
- Inspect the electrode for any scratches or cracks before use.
- Electrodes should be maintained according to manufacturer's recommendations.
- pH meters must be standardized before each use with buffer solutions.

3.10 High Tech Instruments

Apera Instruments PC820 Precision Bench top pH/Conductivity multipara meter Kit (Figure 5)



Figure 5: Benchtop pH/Conductivity Multiparameter Meter Kit

3.11 Checklist for testing of pH

Serial #	Tasks to be performed	Yes/ no
1	Introduction to the topic	
2	Instrument demonstration	
3	Reagents preparation	
4	Testing of water sample	
5	Quality control	
6	Reporting the results	

3.12 Planned Demonstration

Testing of pH in Chemical Lab (Reagents preparation, glassware washing, standards preparation, testing, quality control, calculations and reporting, units conversion)

3.13 Class Exercises

- surprised quiz
- handling of instrument
- analysis of unknown sample

3.14 Group project:

Water Quality Assessment

4 Turbidity

4.1 Introduction

Turbidity is a measure of the clarity of the water. It is the amount of solids suspended in the water. The turbidity of sample is the reduction of transparency due to the presence of Particulate matter such as clay or slit, finely divided organic matter, plankton or other microscopic organisms. These cause light to be scattered and absorbed rather than transmitted in straight lines through the sample. The values are expressed in nephelometric turbidity units (NTU). The method is applicable to drinking, surface and saline waters in the range of turbidity 0-40 NTU. Higher values may be obtained by dilution of the sample (Figure 6)

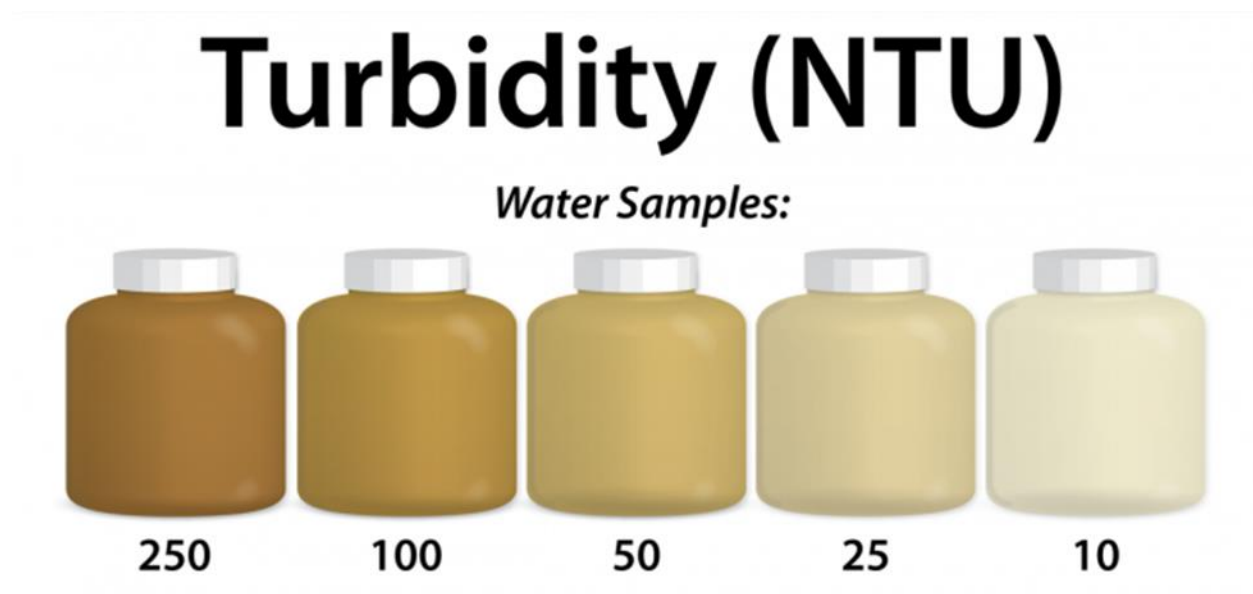


Figure 6: Turbidity levels

4.1.1 Impact of Turbidity

High turbidity can significantly reduce the aesthetic quality of lakes and streams, having a harmful impact on recreation and tourism. It can increase the cost of water treatment for drinking and food processing. It can harm fish and other aquatic life by reducing food supplies.

4.1.2 Ways to remove turbidity

Turbidity can be removed by using different strategies like cloth filtration, sand filtration, settling and decanting, alum flocculation etc.

4.1.3 Permissible limits of turbidity

- Natural water (PSQCA/NSDWQ, 2010): ≤ 5
- Bottled water (PSQCA 2010): 0.5

4.2 Standard Operating Procedure for Analysis of Turbidity

4.2.1 Testing method

Nephelometric method

4.2.2 Principle

It is based on comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension under the same conditions.

4.2.3 Interferences

Colored solutes cause lowering of turbidity values.

4.2.4 Apparatus

- Sample tubes - The sample tubes should be of clear and colorless glass.

4.2.5 Testing Instrument

Turbidity Meter (HANNA, HI 93703) (Figure 7)



Figure 7: Turbidity meter (HANNA 93703)

4.3 Standards and Reagents

a) *Preparation of 400 FTU Stock Turbidity Solution*

- Hydrazine Sulphate (NH_2) H_2SO_4
- Hexa methylene Tetra Amine (CH_2) N_4
- Distilled water

b) *Procedure*

Solution-1: Weigh and dissolve 1g (NH_2) H_2SO_4 in distilled water Transfer to 100 ml volumetric flask and dilute up to 100 ml with distilled water.

Solution-2: Weigh and dissolve 1g (CH_2) N_4 in distilled water and dilute to 100 ml in volumetric flask.

- In 100 ml volumetric flask mix 5 ml of solution (1) and 5 ml of solution 2
- Let it stands for 24 hours at $25 \pm 30^\circ\text{C}$.
- Dilute to mark and mix with distilled water at $25 \pm 30^\circ\text{C}$.

4.3.1 Preparation of Turbidity Working Standard (10 FTU)

a) *Reagents*

- 400 FTU stock Turbidity solution
- Distilled water

Procedure

- Take 2.5 ml stock Turbidity solution in to 100 ml volumetric flask.
- Dilute to 100 ml with Turbidity free water (0 ± 0.02 NTU).
- Transfer to an appropriately labeled bottle
- Standards of 400 FTU & 10FTU
- Distilled Water

4.3.2 Calibration Procedure

- Turn the meter on and wait for the display
- Press the “CAL” key twice at a time. The instrument is now in calibration mode and “CAL” will appear on the lower part of the display
- The calibration date can be edited by simple pressing the “DATE/Key”
- To enter the exact date, press the “READ/key”
- The default blinking parameter is the month on

- The left hand display “(MM.DD)”
- Press the “CAL” key once to go into calibration mode
- A blinking “ZERO” message will appear.
- Take the zero FTU standard and fill the measurement cuvette and insert the cuvette into the measurement cell and press the “CAL” key
- Blinking “SIP” message will appear.
- After 5 seconds the instrument will ask for the 10 FTU standard by displaying “10.0” Repeat the same procedure with 10 FTU standard solution
- Fill the measurement cuvette and insert the cuvette into measurement cell and press the “CAL” key again
- After 50 second the LCD will display. Now calibration is completed and instrument is ready for analysis.

4.4 Analysis Procedure

- Turn the meter on by pressing ON/OFF key.
- Now LCD will change to measurement mode by displaying --.
- Rinse the cuvette with turbidity free water and shake the sample bottle gently.
- Fill the cuvette up to the mark with thoroughly agitated sample.
- Wipe the cuvette thoroughly with lint free tissue, to make the cuvette free of oil drift and fingerprints.
- Shake vigorously.
- Place the cuvette into the cell, the mark on the cuvette cap should point towards the LCD.
- Press the READ key and LCD will display blinking SIP.
- The turbidity value will appear after 25 seconds.

4.5 Expression of Results

The turbidity of sample is measured in NTU

4.5.1 Interpretation of Results

Compare the results with Water Quality Standards or Guidelines to predict the safe or unsafe level.

4.6 Quality Control

- Turbidity Meter is standardized by running known standards.
- Method blank is analyzed after every 10 samples

- Laboratory Control samples of 10 FTU/NTU analyzed with every batch of 10 samples

4.7 Precautions

- Rinse the cuvette with turbidity free water carefully.
- Allow the sufficient time for bubbles to escape before securing the cap and do not over tighten the cap.
- To obtain the sample, shake it thoroughly
- Each time the cuvette is used, tighten the cap to the same degree.
- Presence of debris and coarse sediments which settle out rapidly on the cuvette will give false results.
- Dirty glassware could also affect readings along with scratched or edged vials.

4.8 High Tech instrument for turbidity analysis:

pH / DO/ Conductivity/ TDS /Turbidity Sensor (Figure 8)



Figure 8: pH / DO/ Conductivity/ TDS /Turbidity Sensor

4.9 Checklist for testing of pH

Serial #	Tasks to be performed	Yes/ no
1	Introduction to the topic	
2	Instrument demonstration	
3	Reagents preparation	
4	Testing of water sample	
5	Quality control	
6	Reporting the results	

4.10 Planned Demonstration

Testing of pH in Chemical Lab (Reagents preparation, glassware washing, standards preparation, testing, quality control, calculations and reporting, units conversion)

4.11 Class Exercises

- Surprised quiz and handling of instrument
- analysis of unknown sample

4.12 Group project

Water Quality Assessment

Section B: Chemical Parameters

5 Sodium

5.1 Introduction

Sodium is a common element in the natural environment and is often found in food and drinking water. Sodium ranks sixth among the elements in order of abundance and is present in most natural water. In drinking water, sodium can occur naturally or by the result of road salt application, water treatment chemicals or ion-exchange water-softening units. Sodium levels may also vary in bottled water and carbonated water, depending on the brand.

5.1.1 Source of sodium in drinking water

Sodium and chloride occur naturally in water as a result of erosion or salt water intrusion (when salt water from the ocean seeps into underground water supplies). Sodium may reach both ground and surface water supplies as a result of residential, commercial and industrial activity, such as road salting. Saline intrusion, mineral deposits, seawater spray, sewage effluents, and salt used in road de-icing can all contribute significant quantities of sodium to water.

5.1.2 Importance of sodium for human body

The human body needs sodium in order to maintain blood pressure, control fluid levels and for normal nerve and muscle function. Sodium in drinking water is not a health concern for most people but may be for someone with specific health issues like,

- Persons who are monitoring their salt intake for high blood pressure
- Persons with cardiovascular or heart disease
- Persons with kidney problems
- Persons on low sodium diets.

Excessive intake of very high doses of sodium (accidental poisoning) may cause acute effects such as nausea, vomiting, inflammatory reaction in the gastrointestinal tract, thirst, muscular twitching, convulsions, and possibly death. For long-term lower level exposures, the health effect of primary concern is essential hypertension (high blood pressure).

5.1.3 Ways to reduce high level of sodium in drinking water

Sodium (salt) cannot be easily removed from drinking water and cannot be removed through boiling or conventional filtrations however reverse osmosis, ion exchange or distillation systems can reduce sodium levels but these systems may be expensive to operate.

5.1.4 Permissible limits of sodium

- Natural drinking water (PSQCA/NSDWQ, 2010): NGVS
- Bottled water (PSQCA 2010): 50 ppm

5.2 Standard Operating procedure for analysis of Sodium

5.2.1 Method

Flame Photometric Method

5.2.2 Scope

This standard prescribes method for determination of sodium by flame emission photometric method using flame photometry method.

5.2.3 Sampling & Storage

Acidify the sample to pH 2 with concentrated nitric acid if sample has to analyze after a long time. Store all samples/stock solutions in polyethylene bottles.

5.3 Principle

A flame photometer measures photo electrically the intensity of color imparted to the flame of a burner where the sample is introduced into the flame under carefully standardized conditions. The intensity of color is proportional to the sodium content in the sample. Sodium is determined at a wavelength of 589nm. (Figure 9)

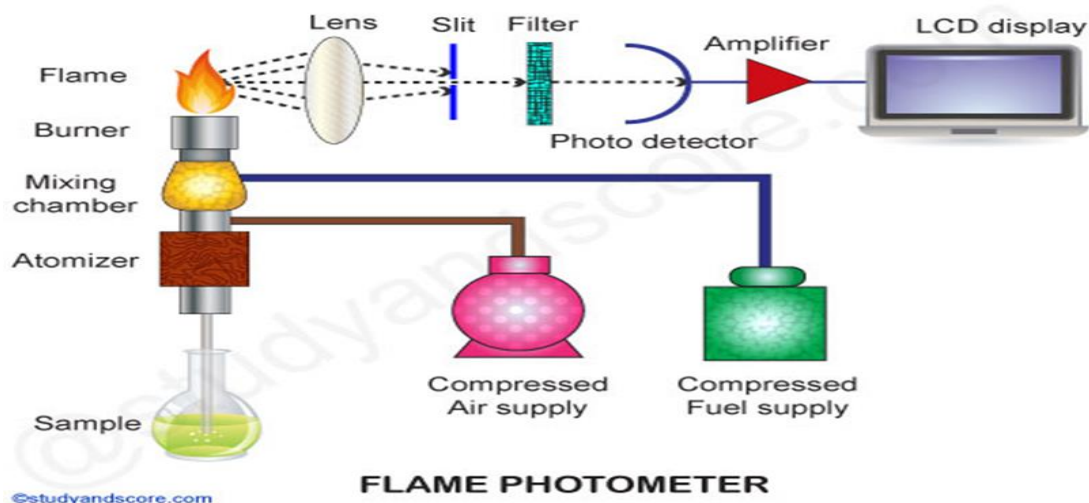


Figure 9: Working Principal of flame photometer

5.4 Interferences

Interference is caused by elements other than those being determined is the chief contributing factor for error in flame photometry. Suspended matter which may interfere mechanically by clogging the burner shall be removed by filtration prior to the analysis.

5.5 Apparatus

5.5.1 Flame photometer:

Jenway PFP 7 (Figure 10)

5.5.2 Glassware

Rinse all glassware's with dilute nitric acid (1:15) followed by several Portions of deionized distilled water.



Figure 10: Flame photometer (JENWAY PFP 7)

5.6 Standards and Reagents

- Sodium Stock Solution (1000 ppm)
- Standard Solutions (by dilution)

a) 1000 ppm Sodium Stock Solution

5.6.1 Reagents

- Sodium Chloride granules
- Distilled water
- Hot air oven

5.6.2 Procedure

- Weigh 2.5434g sodium chloride (dried at 110°C) into 1000 ml volumetric flask.
- Dissolve it by adding little quantity of water.
- Dilute to mark with distilled water.
- Transfer to reagent bottle and label it.

b) Sodium Working Standard

5.6.3 Reagents

- 1000 ppm sodium chloride solution
- Distilled water

5.6.4 Procedure

- Take 25 ml of 1000 ppm sodium chloride solution into 250 ml volumetric flask to prepare 100 ppm of sodium chloride stock solution.
- Dilute to mark with distilled water.
- Check concentration of the standard on flame photometer
- Prepare sodium solution of 10, 20, 30, 40, 50 and 60 ppm by dilution of stock as working standard. (Table 2)

Table 2: Series of calibration standard (Sodium)

Na Calibration standards (ppm)	Volumetric Flask (ml)	Aliquot of 100 ppm Na (ml)	Aliquot of 1000 ppm Na (ml)
10	100	10	1
20	100	20	2
30	250	-	7.5
40	250	-	10
50	250	-	12.5
60	250	-	15

If concentration displayed on the flame photometer matches with the prepared concentration transfer to reagent bottle and label.

5.7 Calibration of Flame Photometer

- After turning on the fuel supply Meter is turned “ON” by pressing power switch.
- Fuel supply is opened by turning FUEL and ignition switch is pressed until “FLM” indicator display.
- The flame is adjusted by rotating knob of fuel adjust.
- Filter for Na/ K is selected by rotating FILTER SELECT.
- 10 ml deionized water is aspirated and its suction is noted per minute.
- If the consumption rate is between 2 and 6 ml/min. then no further action is required
- Standard solution is then aspirated and its value is then adjusted by “FINE CONTROL” until positive reading obtained.
- For potassium standard, D.P switch is turned to one decimal place.
- When blank and standard are set. Unknown samples can be aspirated and the results noted.

5.7.1 Testing

STEP 1 Starting

The fuel supply is turned on at the source.

The meter is turned “ON” by pressing power switch.

STEP 2 Flame adjustments

The ignition switch is pressed meanwhile open fuel by rotating fuel knob anticlockwise.

The ignition switch is pressed until “FLM” indicator displayed on the screen.

The flame is adjusted by rotating knob of fuel adjuster.

STEP 3 Filter Selection

For the determination of sodium turned the FILTER SELECT knob to sodium filter.

The suction of photometer is checked by aspirating 10 ml deionized water per minute.

If it is 2-6 ml then it is OK.

STEP 4 Selection of Standard

Standard solution is aspirated and adjusted by rotating the fine control knob until positive reading obtained. When blank and standard are set. Unknown samples aspirated and the results noted. Number of replicates to be analyzed is “Three” for each sample.

5.8 Quality Control

- Flame photometer is standardized by running blank and standard solutions.
- Method blank is analyzed after every sample
- Laboratory Control samples of 20ppm, 40ppm and 50ppm analyzed with every batch of 10 samples.
- NWQL check repeatability by reanalyzing a pre-analyzed sample once a day
- To ensure the accuracy and precision of method spiking with known standard solution done once in month.
- Known standards are prepared in the NWQL are before and after calibration.
- Standard runs after every 2 samples for accurate results.
- Results of laboratory control sample are regularly displayed in the form of control charts.

5.9 Expression of Results

The sodium is measured in mg/l.

5.9.1 Interpretation of Results

Compare the results with Water Quality Standards or Guidelines to predict the safe or unsafe level.

5.10 Checklist for testing of sodium

Serial #	Tasks to be performed	Yes/ no
1	Introduction to the topic	
2	Instrument demonstration	
3	Reagents preparation	
4	Testing of water sample	
5	Quality control	
6	Reporting the results	

5.11 Planned Demonstration

Testing of Na in Chemical Lab (Reagents preparation, glassware washing, standards preparation, testing, quality control, calculations and reporting, units conversion)

5.12 Class Exercises

- surprised quiz
- handling of instrument
- analysis of unknown sample

5.13 Group project

Water Quality Assessment

6 Potassium

6.1 Introduction

Potassium is a chemical element with the symbol K and atomic number 19. The American Heart Association defines potassium as an electrolyte and nutrient. Potassium is an essential element in humans and is seldom, if ever, found in drinking water at levels that could be a concern for healthy humans.

6.1.1 Sources of potassium

The primary source of potassium for the general population is the diet, as potassium is found in all foods, particularly vegetables and fruits. Resulting levels of potassium in drinking-water are relatively low compared with levels resulting from the use of water softeners using potassium chloride.

6.1.2 Health benefits

It is important to note that potassium is an important nutrient we all need for good health. But, the human body doesn't produce potassium, so we must get it from our diet. Potassium works with sodium to maintain the body's water balance and is also involved in nerve function, muscle control and blood pressure. In some countries, potassium chloride is being used in ion exchange for household water softening in place of, or mixed with, sodium chloride, so potassium ions would exchange with calcium and magnesium ions.

6.1.3 Adverse health effects

Currently, there is no evidence that potassium levels in municipally treated drinking-water, even water treated with potassium permanganate, are likely to pose any risk for the health of consumers. It is not considered necessary to establish a health-based guideline value for potassium in drinking-water, but due to high potassium consumption from drinking-water are chest tightness, vomiting, diarrhea, hyperkalemia (high potassium level in blood), shortness of breath and heart failure.

Ways to remove high level of potassium from drinking water

Potassium may be removed from drinking water by means of reverse osmosis.

6.1.4 Permissible limits of potassium

- Natural drinking water (PSQCA/NSDWQ, 2010): NGVS
- Bottled water (PSQCA 2010): 10

6.2 Standard Operating Procedure for analysis of Potassium

6.2.1 Testing method

Flame Photometric Method

6.2.2 Standards and Reagents

- Stock Potassium Solution (1000 ppm)
- Standard Solutions (by dilution)

a) Stock solution of Potassium (1000 ppm)

6.2.3 Reagents

- Potassium Chloride granules
- Distilled water
- Hot air oven

6.2.4 Procedure

- Weigh 1.907 g KCl dried at 110°C.
- Dissolve it by adding little quantity of water.
- Dilute to mark with distilled water
- Transfer to reagent bottle and label it.

b) Working Standard of Potassium

6.2.5 Reagents

- 1000 ppm Potassium Solution
- Distilled water

6.2.6 Procedure

- Take 25 ml of 1000 mg/l potassium solution in 250 ml flask.
- Dilute to mark with distilled water.
- Check its concentration by means of flame photometer.

- Similarly prepare potassium solution of 2, 05, 10, 15, and 20 ppm by dilution of stock or working standard.
- Follow the Table-3 to prepare the series of calibration standards

Table 3: series of calibration standards

Potassium standards (ppm)	Volumetric Flask (ml)	Aliquot of 100ppm K (ml)
02	250	05
05	250	12.5
10	250	25
15	250	37.5
20	100	20

- If concentration detected by flame photometer matches with the prepared concentration transfer to reagent bottle and label it.

STEP 1 Starting

- The fuel supply is turned on at the source.
- The meter is turned “ON” by pressing power switch.

STEP 2 Flame adjustments

- The ignition switch is pressed meanwhile open fuel by rotating fuel knob anticlockwise.
- The ignition switch is pressed until “FLM” indicator displayed on the screen.
- The flame is adjusted by rotating knob of fuel adjuster.

STEP 3 Filter Selection

- For the determination of potassium turned the FILTER SELECT to potassium filter
- The suction of photometer is checked by aspirating 10 ml deionized water per minute.
- If it is 2-6 ml then it is OK.

STEP 4 Selection of Standard

- Standard solution is aspirated and adjusted by rotating the fine control knob until positive reading obtained.
- For potassium standard, D.P knob is turned to one decimal point.
- When blank and standard are set. Unknown samples aspirated and the results noted.
- Number of replicates to be analyzed are “Three” for each sample.

6.3 Quality Control

- Flame photometer is standardized by running blank and standard solutions.
- Method blank is analyzed after every ten samples
- Laboratory Control samples of 2ppm, and 5ppm analyzed with every batch of 10 samples.
- NWQL check repeatability by reanalyzing a pre-analyzed sample once a day
- To ensure the accuracy and precision of method spiking with known standard solution done once in month.
- Daily quality control data is recorded in QC Data sheet-I.
- Known standards are prepared in the NWQL are before and after calibration.
- Standard runs after every 2 samples for accurate results.
- Results of laboratory control sample are regularly displayed in the form of control charts.

6.4 Expression of Results

The Na/K concentration is measured in mg/l.

6.5 Interpretation of Results

Compare the results with Water Quality Standards or Guidelines (See Annexure-1) to predict the safe or unsafe level

6.6 Precautions

- Do not leave the instrument running unattended while the flame is alight.
- The top of instrument chimney unit becomes very hot when running don't touch it. Never attempt to look down the chimney whilst the flame is running. Always use the inspection window.
- If high concentration salt solution is aspirated for longer periods then deionized water aspirate prior to shut down.

- Take care when preparing standards.
- Always sample from top half of the sample. The bottom half may contain sediments that could block fine tubing.

6.7 Alternate Testing

High tech instrument for sodium and potassium analysis on Inductive Coupled Plasma Spectrophotometer (ICP) (Figure 11)



Figure 11: Inductive Coupled Plasma Spectrophotometer (ICP)

6.8 Checklist for testing of potassium

Serial #	Tasks to be performed	Yes/ no
1	Introduction to the topic	
2	Instrument demonstration	
3	Reagents preparation	
4	Testing of water sample	
5	Quality control	
6	Reporting the results	

6.9 Planned Demonstration

Testing of potassium in Chemical Lab (Reagents preparation, glassware washing, standards preparation, testing, quality control, calculations and reporting, units conversion)

6.10 Class Exercises:

- surprised quiz
- handling of instrument
- analysis of unknown sample

6.11 Group project:

Water Quality Assessment

7 Total and Free Chlorine

7.1 Definition

Chlorine is used as a disinfected agent for the treatment of drinking water supplies. The purpose of this method is to characterize the quality of portable water and to estimate the permissible amount of chlorine in water as its low concentration is not effective for treatment of water and its overdose is carcinogenic.

The chlorination of water supplies and polluted waters serves primarily to destroy or deactivate disease-producing micro-organisms. A secondary benefit, particularly in treating drinking water, is the overall improvement in water quality resulting from the reaction of chlorine with ammonia, iron, manganese, Sulphide and some organic substances.

7.1.1 Total Chlorine

Total chlorine is the sum of free and combined chlorine. When chlorinating most potable water supplies, total chlorine is essentially equal to free chlorine because the concentration of ammonia or organic nitrogen compounds (needed to form combined chlorine) will be very low. When chloramines are present in a municipal water supply, total chlorine will be higher than free chlorine.

7.1.2 Free Chlorine

Free chlorine is defined as the concentration of residual chlorine in water present as dissolved gas (Cl_2), hypochlorous acid (HOCl), and/or hypochlorite ion (OCl^-).

7.1.3 Combined Chlorine

Combined chlorine is defined as the residual chlorine existing in water in chemical combination with ammonia or organic amines which can be found in natural or polluted waters. Ammonia is sometimes deliberately added to chlorinated public water supplies to provide inorganic chloramines. This process is generally referred to as “chloramination”.

7.1.4 Chlorine demand

Chlorine demand is the difference between the amount of chlorine added to water or wastewater and the amount of residual chlorine remaining after a given contact time. Chlorine demand may change with dosage, time, temperature, pH, and nature and amount of the impurities in the water.

Chlorine Demand = Chlorine Applied – Chlorine Residual

7.1.5 Adverse effects of chlorination

Chlorine poisoning can occur when you touch, swallow, or inhale chlorine. Chlorine reacts with water outside of the body and on mucosal surfaces inside your body including the water in your digestive tract causing hydrochloric acid and hypochlorous acid to form. Both of these substances can be extremely poisonous to humans.

7.1.6 Interference

Color and turbidity interfere with the results of chlorine. Organic contaminants may produce a false free chlorine reading in most colorimetric methods.

7.2 Testing Method

DPD Method

7.2.1 Testing Instrument

Chlorine Meter (Merck) (Figure 12)



Figure 12: Chlorine testing kit (Merck)

7.3 Principle of Measurement

Chlorine in aqueous solution (water) is not stable. The chlorine content of water will decrease rapidly as exposure of sunlight or agitation accelerates the reduction of

chlorine therefore start chlorine determination as soon as sample is received by using commercially available reagents.

7.3.1 Standards & Reagents

- Deionized water – free of Cl_2 .
- 7.2 N,N-Di-n-propyl-1,4-phenylenediamine (Spectroquant® Reagent Cl_2 -1).
- Potassium Iodide solution (Spectroquant® Reagent Cl_2 -2)
- Potassium Permanganate, ACS grade crystals.
- Stock Potassium Permanganate Solution: Place 0.891 g KMnO_4 in a volumetric flask and dilute to 1 liter.
- Standard Potassium Permanganate Solution: Dilute 10.00 mL of stock potassium permanganate solution (7.3 above) to 100 mL with distilled water in a volumetric flask. One mL of this solution diluted to 100 mL with distilled water is equivalent to 1.00 mg/L Chlorine.

7.3.2 Procedure for analysis of free chlorine

- Check the pH of samples, and if necessary, adjust to pH between four and eight.
- With a volumetric pipette, add 6.0 ml of sample to a test tube.
- Add one level blue micro spoon of reagent Cl_2 -1.
- Shake the tube vigorously to dissolve the solid substance.
- Mix well and set aside for one minute

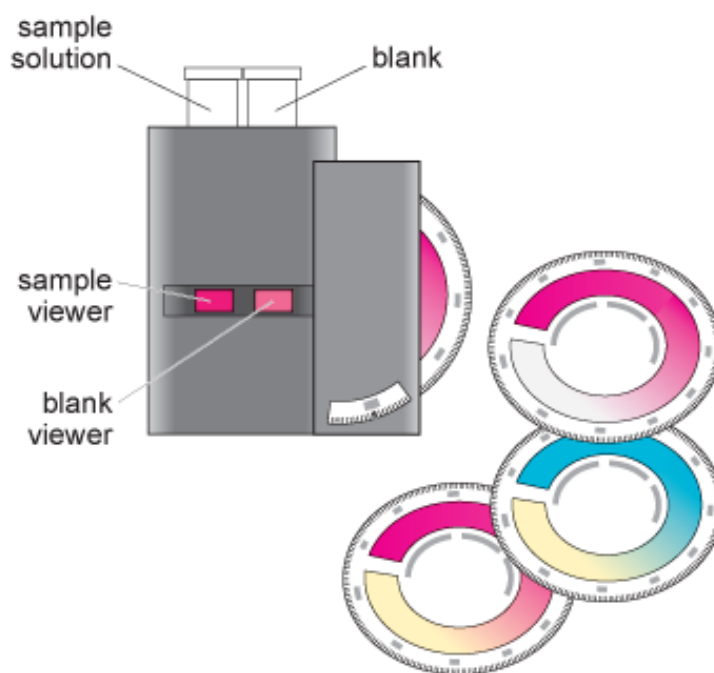


Figure 13: Chlorine Color Comparator Disc

- Compare the color with the color scale (color comparator disc), read off the result of free chlorine in the sample. (Figure 13)

7.3.3 Procedure for analysis of total chlorine

- Check the pH of samples, and if necessary, adjust to pH between four and eight.
- With a volumetric pipette, add 6.0 ml of sample to a test tube.
- Add one level blue micro spoon of reagent Cl2-1
- Shake the tube vigorously to dissolve the solid substance.
- Add 2 drops of reagent Cl2-2
- Shake the tube vigorously to dissolve the solid substance.
- Mix well and set aside for one minute
- Compare the color with the color scale, read off the result of total chlorine in the sample.

7.4 Expression of Results

The chlorine of sample is measured in mg/l.

7.4.1 Interpretation of Results

Compare the results with Water Quality Standards or Guidelines to predict the safe or unsafe level.

7.5 Quality Control

- Method blank is analyzed after every 10 samples.
- Laboratory Control samples analyzed with every batch of 10 samples

7.6 Precautions

- Do not touch the cuvette walls with hands.
- Do not let the test sample stand too long after reagent is added or accuracy will be lost.
- Whenever the cuvette is placed into the measurement cell, it must be free of fingerprints, oil or dirt.
- To obtain the representative sample, gently but thoroughly, mix it before sample is taken.
- Each time the cuvette is used, tighten the cap to the same degree.

7.7 High Tech Instrument

- High Accuracy Portable PH Chlorine Digital Tester TDS Meter (Figure 14)



Figure 14: Portable PH Chlorine Digital Tester TDS Meter

7.8 Checklist for testing of chlorine

Serial #	Tasks to be performed	Yes/ no
1	Introduction to the topic	
2	Instrument demonstration	
3	Reagents preparation	
4	Testing of water sample	
5	Quality control	
6	Reporting the results	

7.9 Planned Demonstration

Testing of chlorine in Chemical Lab (Reagents preparation, glassware washing, standards preparation, testing, quality control, calculations and reporting, units conversion)

7.10 Class Exercises

- surprised quiz
- handling of instrument
- analysis of unknown sample

7.11 Group project

Water Quality Assessment