**DETERMINATION OF pH**

**Apparatus required:**

Glass electrode, Reference electrode (mercury/calomel or silver/silver chloride) and pH meter

**Procedure:**

Electrometric method: pH is determined by measuring the Electro Motive Force (E.M.F) of a cell comprising an indicator electrode (an electrode responsive to hydrogen ions such as a glass electrode) immersed in the test solution and the reference electrode (usually a mercury/calomel electrode). Contact between the test solution and the reference electrode is usually got by means of a liquid junction, which forms a part of reference electrode. E.M.F of this cell is measured with pH meter, that is, a high impedance voltmeter calibrated in terms of pH. The electrode is allowed to stand for 2 minutes to stabilize before taking reading for reproducible results (at least ±0.1 pH units).

**DETERMINATION OF ALKALINITY OF WATER**

**Apparatus:**

1. Burette 2. Pipette 3. Erlenmeyer flasks 4. Indicator solutions

Reagents:

1. Standard Sulphuric Acid ( 0.02 N)

2. Phenolphthalein indicator

3. Methyl orange indicator

**Procedure:**

Phenolphthalein Alkalinity:

1. Take 25ml of sample in a conical flask.

2. Add 3-4 drops of phenolphthalein indicator. If the pH of sample is above 8.3, sample turns

pink.

3. Titrate with 0.02 N H2SO4 in a burette till the colour disappears.

4. Note down the volume of H2SO4 added (V1)

Total Alkalinity:

1. Take 25ml of sample in a conical flask.

2. Add 3-4 drops of Methyl orange indicator. The sample turns yellow.

3. Titrate it against 0.02N H2SO4 till the colour of the sample turns orange.

4. Note down the total volume of H2SO4 added (V2)

**Calculation:**

1. Phenolphthalein alkalinity in mg/L as CaCO3 =

𝑉∗𝑁∗50∗1000

𝑉𝑜𝑙𝑢𝑚𝑒 𝑜𝑓 𝑠𝑎𝑚𝑝𝑙𝑒 𝑡𝑎𝑘𝑒𝑛

2. Total alkalinity in mg/L as CaCO3 = 𝑉∗𝑁∗50∗1000/

𝑉𝑜𝑙𝑢𝑚𝑒 𝑜𝑓 𝑠𝑎𝑚𝑝𝑙𝑒 𝑡𝑎𝑘𝑒

**DETERMINATION OF TURBIDITY**

**Procedure:**

1. Switch on the instrument and allow sufficient warm-up period.

2. Take distilled water or bank solution in the test tube holder and close the lid. Make sure that

the mark on the test tube coincides with mark on the panel.

3. Select required range for measurement.

4. Adjust the displayed to ‘000’ by adjusting set zero knob.

5. Remove the test tube containing distilled water and insert another test tube containing standard

solution (say 100 NTU or 400 NTU). Place it in test tube holder.

6. Adjust the calibrate knob so that the display reach the standard solution value.

7. Again check ‘0’ display with distilled water. The instrument is now calibrated.

8. Place the given sample whose turbidity is to be determined in the test tube and take the reading

in NTU.

**DETERMINATION OF TOTAL SOLIDS**

**Apparatus:**

Evaporating dishes – 100 ml porcelain dish, steam bath, drying oven, desiccators, and Electronic balance and measuring jars.

**Procedure**:

The sum of weights of Total Suspended Solids(TSS) and Total Dissolved Solids(TDS) in same units.

(OR)

A known volume of the well-mixed sample (50ml) is measured into a pre-weighed dish and evaporated to dryness at 103 o C on a steam bath. The evaporated sample is dried in an oven for about an hour at 103-105 o C and cooled in desiccators and recorded for constant weight.

**Calculation:**

Total solids (mg/l)   =  (W1 - W2) (1000)

Sample volume (ml)

Where, W1    = Weight of dried residue + dish,

W2 = Weight of empty dish,

V= Volume taken.

**DETERMINATION OF TOTAL SUSPENDED SOLIDS**

**Apparatus:**

Porcelain dish (100ml capacity), Watt man filter paper, flasks, measuring jar, drying oven and filtration apparatus.

**Procedure:** The known volume of vigorously shaken sample (50ml) is filtered by filter paper into a pre-weighed Porcelain dish, and Filter Paper is dried for an hour at 103-105o C in an oven, cooled in desiccator and weighed for constant weight.

**Calculation:**

Total Suspended Solids  (mg/l)  =  (W1 - W2) x (1000)

Sample volume (ml)

W1    = Weight of dried glass fibre filter + residue,

W2 = Weight of glass fibre filter disk before filtering.

**DETERMINATION OF CHLORIDES**

**Reagents:**Potassium chromate indicator,Silver nitrate solution (0.014N)

**Procedure:**

A known volume of filtered sample (50ml) is taken in a conical flask, to which about 0.5ml of potassium chromate indicator is added and titrated against standard silver nitrate till silver dichromate (AgCrO4) starts precipitating.

**Calculation:**

Chlorides (Cl-) =   (A-B) x (N) x (35.45)

Sample taken in ml

Where, A - Volume of silver nitrate consumed by the sample

B - Volume of silver nitrate consumed by the blank

N - Normality of silver nitrate

**DETERMINATION OF CONDUCTIVITY**

**Apparatus:**

1. Conductivity meter with measuring cell; 2. Beaker 3. Thermometer

Procedure:

Note: If standard KCl solution conductivity is known at different temperature. This

calibration method is preferable.

1. Select COND mode

2. After thoroughly cleaning, immerse the cell in the standard solution say 0.1N KCl

which has specific conductivity of the order of 0.01412 at 300C. (=14.12µs)

3. Select 20ms range (select range as per standard KCl solution conductivity).

4. Set conductivity (as per temperatures) using CELL constant POT and then lock

using lock nut. Now calibration is over.

**CALIBRATION** USING COND (25 0C) MODE:

Note: If standard KCl solution conductivity is not known at different temperature (only

at 250C specific conductivity known). This calibration method is preferable.

1. Select TEMP mode

2. Set temperature of standard solution using TEMP

3. Select COND (250C) mode

4. After thoroughly cleaning, immerse the cell in the standard solution, say 0.1N KCL (aqueous) which

has specific conductivity of the order of 0.01288 = (12.88µs) at 250C

5. Select 20ms range.

6. Set conductivity (as per 250C temperature) using CELL constant POT and then lock using lock nut.

Now calibration is over.

RESULT: The electrical conductivity of given water sample is ---------------- µhoms/cms

**TOTAL HARDNESS OF WATER**

**Procedure** for calculation of hardness of water by EDTA titration

Take a sample volume of 20ml (V ml).

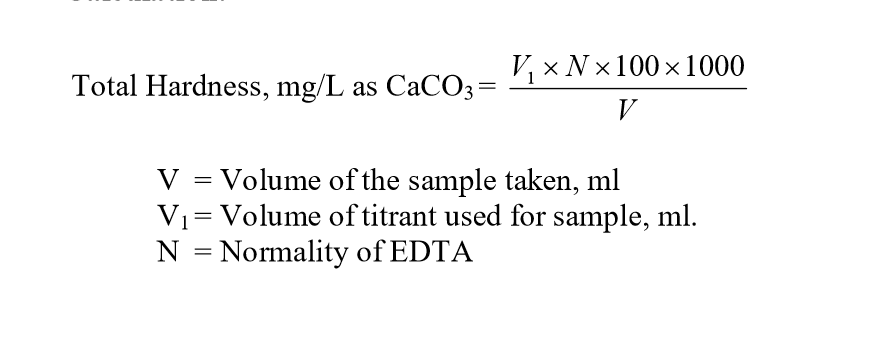
Dilute 20ml of the sample in Erlenmeyer flask to 40ml by adding 20ml of distilled water.

Add 1 mL of ammonia buffer to bring the pH to 10±0.1.

Add 1 or 2 drops of the indicator solution. If there is Ca or Mg hardness the solution turns wine red.

Add EDTA titrant to the sample with vigorous shaking till the wine red colour just turns blue.

Note the volume of titrant added (V1 ml).



**Determination of permanent hardness of water by EDTA method:**

The temporary hardness can be removed by boiling. The permanent hardness is determined first by precipitating the bicarbonates of Ca2+ and Mg2+ by heating and filtering off.

**Procedure:**

1. Take 100ml of water sample into a beaker and boil gently for 15-20 minutes.
2. Cool the solution then filter and wash the precipitate several times. Collect both the filtrate into a 250ml volumetric flask and level up to the mark with distilled water. Then, shake well.
3. Take 50ml of solution from volumetric flask with pipette and place into a conical flask.
4. Add 1-5ml NH4OH/NH4Cl buffer solution. It increases the pH level and should be 10. Check the pH with standardize pH meter.
5. Add 2-3 drops 0.1M Mg-EDTA solution and 3-4 drops Eriochrome Black T indicator. Then, shake well and the color becomes wine red.
6. Fill up the burette with standardized 0.01M EDTA solution. Record the initial burette reading and titrate the water sample with this standard solution.
7. At the end point the color of the solution turns into blue from wine red. Titrate carefully near the end point.
8. Take the final burette reading. Let, it is V1 ml.
9. Repeat the titration process at least three times.
10. You can run a blank titration for more accurate result. Let, it is V2 ml.

**Permanent hardness calculation:**  
In case of blank titration, the calculate volume of EDTA required by sample water, V = (V1-V2)ml  
The permanent hardness can be calculated by using the following formula.  
1ml 0.01M EDTA ≡ 1.00mg CaCO3  
∴ Vml 0.01M EDTA = V ᵡ 1.00mg CaCO3

The 100ml sample water is dilute into a 250ml volumetric flask  
Hence, 50ml dilute water = 100 ᵡ 50/250 ml = 20ml sample water

Now, 20ml of sample water ≡ V ᵡ 1.00mg CaCO3  
∴ 1000ml of sample water ≡ V ᵡ 1.00mg ᵡ 1000/20 CaCO3  
≡ V ᵡ 1.00 ᵡ 50 ppm CaCO3

**Result**  
Amount of permanent hardness present in the given water sample = ppm

**DETERMINATION OF IRON**

**Apparatus:**

1. Nessler’s tubes; 2. Conical flasks; 3. Pipettes; 4. Hot Plate

**Procedure:**

1. Take 50ml of given sample in a conical flask.

2. Add 1 ml of Hydroxyl amine hydrochloride solution and 2ml of Conc. HCl. Add few glass beads and heat this sample until the volume of the solution reduces to 15 – 20 ml.

3. Cool the solution to room temperature and transfer this to a Nessler’s tube.

4. Add 2ml of phenanthroline solution and 10 ml of Ammonium acetate buffer.

5. Make up the contents of Nessler’s tube exactly to 100ml by adding distilled water and allow at least 10-15 minutes for colour development.

6. Compare this solution with Iron standards and note down the reading with which standard it matches.

7. The matching colour standard will give the concentration of iron in the sample.

**DETERMINATION OF CALCIUM HARDNESS**

(i) Take 25- or 50-ml sample in a conical flask

(ii) Add 1 ml NaOH to raise pH to 12.0 and add a pinch of murexide indicator.

(iii) Titrate immediately with EDTA till **pink** colour changes to **purple**. Note the volume of EDTA

used (**A1**).

(iv) Run a reagent blank. Note the ml of EDTA required (**B1**) and keep it aside to compare end

points of sample titrations.

(v) Calculate the volume of EDTA required by sample, **C1** = **A1** – **B1**.

**OBSERVATIONS AND CALCULATIONS**

ii) Calcium hardness as CaCO3, mg /l = **C1 X D** **X** **1000** /

Volume of sample in ml

Where

C1= volume of EDTA used by sample (with murexide indicator)

D = mg CaCO3 equivalent to 1 ml EDTA titrant (1 ml 0.01M EDTA ≡ 1.000 mg CaCO3)

(D = 1 × Molarity of EDTA / )

0.01M

(iii) Magnesium hardness

Magnesium Hardness = Total hardness as CaCO3, mg/l – Calcium hardness as CaCO3, mg/l.

**DETERMINATION OF**

Na

SO4

Organic

Inorganic

Ca & Mg