COMPLETE ANALYSIS OF WATER

Abstract

This study was conducted on determine [Conductivity, Total dissolved solids, total suspended solids, pH] and many elements concentration which determine by titrimetric methods such as (Chlorides, Total hardness, Calcium, Magnesium, Total alkalinity, P-alkalinity) and many elements concentration can be determine by Spectrophotometer (DR-3900) such as (Total and Free Chlorine, Total iron, Sulphate, Potassium, Silica and Manganese) and we can determine physical parameters (Color, Odor and Turbidity) in the sample of water.

Then we can determine the microbial test (Total plate count, E-Coli, Coli-form, Streptococcus and Pseudomonas) in the sample of water.

For wastewater, we can determine of [Dissolved Oxygen (DO), Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD) and Total Organic Carbon (TOC)] in the sample of wastewater.

Finally we can comparison the result of these experiments according to Standard specifications for drinking water to the World Health Organization **(WHO)**

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Standard specifications for drinking water according to the World Health Organization **(WHO)**

Element to be analyzed	Units	Accepted Limited (WHO)	Results
Bacteriological Parameters:- ✓ Type of water	---	---	
✓ Odor	---	Odorless	
✓ Total Plate Count at 37 ° C	CFU/ml	25 CFU/ml	
✓ Coli-form	---	Zero	
✓ E-Coli	---	Zero	
✓ Pseudomonas	---	Zero	
✓ Streptococcus	---	Zero	
Physical Parameters:- ✓ Color	mg/L Pt Co	Colorless	
✓ Turbidity	NTU	< 1	
Physiochemical Parameters:- ✓ Conductivity	µs / cm	< 1200	
✓ Total Dissolved Solids [TDS]	mg/L	< 1000	
✓ pH	---	6.8 – 8.5	
✓ Total Hardness $[CaCO_3]$	mg/L	< 500	
✓ Calcium $[Ca^{++}]$	mg/L	< 140	
✓ Magnesium $[Mg^{++}]$	mg/L	< 36	
✓ Sodium $[Na^{+}]$	mg/L	< 200	
✓ Potassium $[K^{+}]$	mg/L	< 10	
✓ Chloride $[Cl^{-}]$	mg/L	< 250	
✓ Sulfate $[SO_4^{--}]$	mg/L	< 250	
✓ Silicate $[SiO_2^{--}]$	mg/L	---	
✓ Iron $[Fe^{++}]$	mg/L	< 0.3	
✓ Manganese $[Mn^{++}]$	mg/L	< 0.4	
✓ Total Alkalinity $[CaCO_3]$	mg/L	< 120	
✓ Bicarbonate Alkalinity$[HCO_3]$	mg/L	---	
✓ Hydroxide Alkalinity $[OH^{-}]$	mg/L	---	
✓ Total Chlorine	mg/L	< 5	
✓ Free Chlorine	mg/L	< 5	

For wastewater

Element to be analyzed	Units	Accepted Limited (WHO)	Results
- Dissolved Oxygen (DO)	mg/L		
- Biochemical Oxygen Demand (BOD)	mg/L		
- Chemical Oxygen Demand (COD)	mg/L		
- Total Organic Carbon (TOC)	mg/L		
- Oil and Grease	mg/L		

PHYSIOCHEMICAL PARAMETERS:-

Conductivity

Measure by Myron (4P or 6P), HANNA

Unit: - µs / cm

Total Dissolved Solid (TDS)

Total dissolved solids refer to the filterable residue that pass through a standard filter disk and remain after evaporation and drying to constant weight at (103 - 105℃)

$$TDS = Conductivty * Factor\ (Type\ of\ water)$$

Unit:-PPM (Part Per Million)

Total Suspended Solids (TSS) [1]

Total suspended solids refer to the non-filterable residue retained by a standard filter disk and dried at 103-105℃.

Apparatus: -
Gooch Crucible, Filter paper, Vacuum, Oven, Weight balance

Procedure:-

- Put a filter paper on the bottom of a clean Gooch crucible.
- Take 20 ml of distilled water and Turn on Vacuum, repeat this process two more times.
- Remove crucible to an oven and dry it for 1 hr at 103℃
- After drying, weight the crucible and place it on a suction unit **(A)**
- Take 25 ml of sample and place it in the crucible and Then filter
- After filtration, dry the crucible at 103℃ for 1 hr
- Weight till constant weight is obtained **(B)**
- Calculate Total Suspended Solids from equation :-

$$TSS = \frac{(A - B) * 10^6}{ml\ of\ sample}$$

Where: -
A = weight of residue and crucible, **B =** weight of crucible

pH

$$pH = -\log[H^+]$$

Measure by pH meter, pH electrode, Indicator (Phenol Red), some of device HANNA

TITRIMETRIC METHODS: -

Chloride (Precipitation Reactions)

In this experiment we want to know the Normality of Sliver Nitrate

$$AgNO_3 + NaCl \rightarrow AgCl \downarrow + NaNO_3$$

$$2AgNO_3 + K_2CrO_4 \rightarrow Ag_2CrO_4 \downarrow + 2KNO_3$$

Apparatus: -

Cylinder, Conical flask, Burette, Pipette, Reagent

Procedure 1:-

- Take 50mL of NaCl(0.028 N) to Conical Flask
- Add 10 drop from Potassium chromate Indicator the color become yellow
- Titrate against $AgNO_3$ (0.028 N) until till white precipitate
- Record the Volume of $AgNO_3$ (V1)
- Calculate:-

$$(N * V)_{NaCl} = (N * V1)_{AgNO_3}$$

$$\therefore (N)_{AgNO_3} = \frac{0.028 * 50}{(V1 - 0.2)}$$

Procedure 2:-

- Take 50mL of the sample to Conical Flask
- Add 10 drop from Potassium chromate Indicator the color become yellow
- Titrate against $AgNO_3$ until till Red brown precipitate
- Record the Volume of $AgNO_3$ (V)
- Calculate Chloride from equation:-

$$cl^- (ppm) = \frac{(V - 0.2) * (eq.wt)_{Cl} * (N)_{AgNO_3} * 1000}{(v)_{sample}}$$

Which:-

M.wt of Cl^- = 35.45, **V** = Volume of $AgNO_3$

value of (0.2) = this value is experiment of Blank. (We repeat produce 2 with distilled water)

$$cl^- (ppm) = \frac{(V - 0.2) * (\frac{35.45}{1}) * (N)_{AgNO_3} * 1000}{50}$$

$$\therefore cl^- (ppm) = (V - 0.2) * (N)_{AgNO_3} * 709$$

Total Hardness (Complexiметric Reactions)

$$Total\ Hardness = Calcium\ Hardnees + Magnesium\ Hardness$$

$$CaCO_3 + EDTA^{-4} \rightarrow Ca\ (EDTA)^{-2} + {CO_3}^{-2}$$

$$MgCO_3 + EDTA^{-4} \rightarrow Mg\ (EDTA)^{-2} + {CO_3}^{-2}$$

Apparatus: -

Cylinder, Conical flask, Burette, Pipette, Reagent

Total Hardness as Calcium Carbonate

Procedure:-

- Take 50mL of the sample to Conical Flask.
- Add a few from EBT Indicator. EBT = EriochromBlack T
- Add 5 drop from ammonia Buffer until Violet color.
- Titrate against EDTA (0.01M) until till Blue color.
- Record the Volume of EDTA (V)
- Calculate Total Hardness from equation :-

$$(\boldsymbol{N * V})_{\boldsymbol{sample}} = (\boldsymbol{N * V})_{\boldsymbol{EDTA}}$$

$$(N)_{sample} = wt\ \left(\frac{mg}{l}\right)(ppm) = \frac{(N)_{EDTA} * (V)_{EDTA} * (eq.wt)_{CaCO_3} * 1000}{(V)_{sample}}$$

$$(N)_{sample} = wt\ \left(\frac{mg}{l}\right)(ppm) = \frac{0.02 * (V)_{EDTA} * (\frac{100}{2}) * 1000}{50}$$

$$\therefore \boldsymbol{Total\ Hardness\ as\ CaCO_3 = (V)_{EDTA} * 20}$$

Calcium Hardness as Calcium Carbonate

Procedure:-

- Take 50mL of the sample to Conical Flask
- Add a few from Muroxide Indicator
- Add 1 drop from NaOH (4M) until pink color
- Titrate against EDTA (0.01M) until till Violet color
- Record the Volume of EDTA (V)
- Calculate Calcium Hardness from equation :-

$$(\boldsymbol{N * V})_{\boldsymbol{sample}} = (\boldsymbol{N * V})_{\boldsymbol{EDTA}}$$

$$\therefore \boldsymbol{Calcium\ Hardness\ as\ CaCO_3 = (V)_{EDTA} * 20}$$

But we need Calcium Ion

$$\therefore \boldsymbol{Ca^{++} = Calcium\ Hardness / 2.497}$$

$$Magnesium\ Hardness = Total\ Hardness - Calcium\ Hardness$$

$\therefore$ *we know Magnesium ion as*

$$\boldsymbol{Mg^{++} = 4.118 * [Total\ Hardness - (2.497 * Calcium\ Hardness)]}$$

Total Alkalinity (M-alkalinity) (Acid-Base Reactions)

$$Total\ Alkalinity = [\ HCO_3^{-} + CO_3^{--} + OH^{-}\ ]$$

$$2NaHCO_3 + 2H_2SO_4 \rightarrow 2Na_2SO_4 + H_2O + CO_2 \uparrow$$

Apparatus: -

Cylinder, Conical flask, Burette, Pipette, Reagent

Procedure:-

- Take 50mL of the sample to Conical Flask
- Add 2 drop from Methyl orange Indicator until bill orange
- Titrate against H_2SO_4 (0.02N) until till Red orange
- Record the Volume of H_2SO_4 (V)
- Calculate Total Alkalinity from equation :-

$$\boldsymbol{(N * V)_{sample} = (N * V)_{H_2SO_4}}$$

$$(N)_{sample} = wt\ \left(\frac{mg}{l}\right)(ppm) = \frac{\boldsymbol{(N)_{H_2SO_4} * (V)_{H_2SO_4}} * (eq.wt)_{CaCO_3} * 1000}{(V)_{sample}}$$

$$\therefore \boldsymbol{Total\ Alkalinity\ as\ CaCO_3 = (V)_{H_2SO_4} * 20}$$

Procedure: - P-alkalinity

- Take 50mL of the sample to Conical Flask
- Add 2 drop from Phenol Phthalein Indicator until Pink
- Titrate against H_2SO_4 (0.02N) until till Colorless
- Record the Volume of H_2SO_4 (V)
- Calculate P-alkalinity from equation :-

$$\therefore \boldsymbol{P - alkalinity\ as\ CaCO_3 = (V)_{H_2SO_4} * 20}$$

SPECTROPHOTOMETER: - Hach (DR-3900)

The basic principle of this method is that "each composite absorbs or reflects light through a specific range of wavelengths" where the device measures the amount of absorbed photons.

Free & Total Chlorine

Apparatus: -
Cuvette, Reagent, device DR-3900

Procedure:-

- Take 10mL of the sample and put it in Cuvette
- Push Zero the device DR-3900
- Start a time for 3 minutes
- Remove Cuvette and Put DPD reagent for (Total or Free Chlorine)

  DPD = N,N-Diethyl-P-Phenylenediamine
- Push read, record the results of (Total or Free Chlorine)

We have two reagents (DPD Free Chlorine, DPD Total Chlorine)

Total Iron

Apparatus: -
Cuvette, Reagent, device DR-3900
Procedure:-

- Take 10mL of the sample and put it in Cuvette
- Push Zero the device DR-3900
- Start a time for 3 minutes
- Remove Cuvette and Put Ferrover Iron reagent for (Total Iron)
- Push read, record the results of (Total Iron)

Sulphate

Apparatus: -
Cuvette, Reagent, device DR-3900
Procedure:-

- Take 10mL of the sample and put it in Cuvette
- Push Zero the device DR-3900
- Start a time for 5 minutes
- Remove Cuvette and Put Sulfaver 4 sulfate reagent for (Sulphate)
- Push read, record the results of (Sulphate)

Potassium

Apparatus: -
Conical flask, Cuvette, Reagent, device DR-3900

Procedure:-

- Take 25mL of the sample and put it in Cylinder
- Put Potassium 1 reagent (Powder), potassium 2 reagent (Solution) and Potassium 3 reagent (Powder) in Conical flask
- Take 10mL from Mixing and put it in Cuvette
- Push Zero The device DR-3900 By another Cuvette But this cuvette contain 10mL of pure sample
- Start a time for 3 minutes
- Put the cuvette which contain 10mL from Mixing
- Push read, record the results of (Potassium)

Silica

In this experiment we have two ranges (High range it happens on Feed water but Low range it happens on Product water)

Apparatus: -
Cuvette, Reagent, device DR-3900

Procedure: - High range

- Take 10mL of The sample and put it in Cuvette
- Push Zero The device DR-3900
- Start a time for 10 minutes
- Remove Cuvette and Put Molybdate reagent (Powder) and Acid reagent (Powder) for (Silica_HR)
- Start a time for 2 minutes
- Remove Cuvette and Put Citric Acid reagent (Powder) for (Silica_HR)
- Push read , record the Results of (Silica_HR)

Procedure: - Low range

- Take 10mL of The sample and put it in Cuvette
- Push Zero The device DR-3900
- Start a time for 4 minutes
- Remove Cuvette and Put 14 Point Molybdate reagent (Solution) for (Silica_LR)
- Start a time for 1 minutes

- Remove Cuvette and Put Citric Acid reagent (Powder) for (Silica_LR)
- Start a time for 2 minutes
- Remove Cuvette and Put Amino Acid reagent (Powder) for (Silica_LR)
- Push read, record the Results of (Silica_LR)

Manganese

In this experiment we have two ranges (High range it happens on Feed water but Low range it happens on Product water)

We Push Zero the device DR-3900 by distilled Water, I mean: -

We take 10mL of the distilled water and put it in Cuvette
remove Cuvette and Put Powder for (Manganese _HR) or Powder for (**Manganese _LR)**

Now; We Push Zero the device DR-3900, I can make the experiment

Apparatus: -
Cuvette, Reagent, device DR-3900

Procedure: - High range

- Take 10mL of the sample and put it in Cuvette
- Start a time for 2 minutes
- Remove Cuvette and Put Buffer Powder Citrate and Put Sodium Periodate (Powder) for (Manganese_HR)
- Push read, Record the Results of (Manganese_HR)

Procedure: - Low range

- Take 10mL of the sample and put it in Cuvette
- Start a time for 2 minutes
- Remove Cuvette and Put Ascorbic acid reagent (Powder) ,12 point Alkaline Cyanide reagent (Solution) and Put 12 point Di-solution reagent (Solution) for (Manganese _LR)
- Push Read , Record the Results of (Manganese _LR)

✓ If you want to identify more elements such as nitrate, nitrite, zinc and fluoride, you can download from site Hach.

PHYSICAL PARAMETERS: -

Color (Colorimetric-Platinum-Cobalt) [2]

Color is measured by visual comparison of the sample with platinum-cobalt standards one unit of color is that produced by 1 mg/L platinum in the form of the chloroplatinate ion.

Apparatus:-
Nessler tubes: Matched, tall form, 50mL capacity.

Reagents:-
Standard chloroplatinate solution: Dissolve (1.246 g) potassium chloroplatinate,K_2ptCl_6 , (equivalent to 0.5 g metallic Pt) and (1.0 g) crystalline cobaltous chloride, $CoCl_2.H_2O$, in distilled water containing 100mL of conc. HCl. Dilute to 1000mL with distilled water. This standard solution is equivalent to 500 color units.

Preparation of Standards:-
Prepare standards in increments from 5 to 70 units. The following series is suggested:

ml of Standard Solution Diluted to 50.0 ml Color in with Distilled Water	Chloroplatinate Units
0.0	0
0.5	5
1.0	10
1.5	15
2.0	20
2.5	25
3.0	30
3.5	35
4.0	40
4.5	45
5.0	50
6.0	60
7.0	70

Protect these standards against evaporation and contamination by use of clean, inert stoppers.

Procedure: -

Apparent color: Observe the color of the sample by filling a matched Nessler tube to the 50 ml mark with the water and compare with standards. This comparison is made by looking vertically downward through the tubes toward a white or specular surface placed at such an angle that light is reflected upward through the columns of liquid. If turbidity has not been removed by the procedure given in (8.2), report the color as "apparent color". If the color exceeds 70 units, dilute the sample with distilled water in known proportions until the color is within the range of the standards.

True color: Remove turbidity by centrifuging the sample until the supernatant is clear. The time required will depend upon the nature of the sample, the speed of the motor, and the radius of the centrifuge, but rarely will more than one hour be necessary. Compare the centrifuged sample with distilled water to insure that turbidity has been removed. If the sample is clear, then compare with standard as given in

Calculate the color units by means of the following equation:

$$Color\ Units = \frac{A * 50}{V}$$

Where: -
A = estimated color of diluted sample, **V** (mL) **=** Volume of sample taken for dilution

- **Odor** [3]

Apparatus: -
Glass capacity of 1 liter

Procedure: -
Detect the smell of water in cold temperature

- Take 500mL of the sample
- Put in glass and flipping for 2 - 3 second
- Remove the plug and smell the Odor

Detect the smell of water in hot temperature

- Take 500mL of the sample
- Put in glass and heating for 58 - 60℃ .
- Remove the plug and smell the Odor

- **Turbidity**

Measured by Turbidimeter or Nephelometer
which measures the intensity of light scattered at 90 degrees as a beam of light passes through a water sample

Unit: - NTU = Nephelometric Turbidity Unit, FTU = Formazine Turbidity Unit and JUT = Jackson Turbidity Unit

Formazine $(NH_4)2SO_4$**: -** A polymer suspended in water that causes light dispersion when passing through the solution

MICROBIAL TEST: -

Notes: -

- ✓ We make each microbial test next to the flame
- ✓ Each Petri-dish is sterilized in the oven at 150 ° C for 2 hours

Total Plate Count (NUTRIENT AGAR)

Total plate count can be made using plate count agar

Typically this agar consists of peptone, yeast extract glucose, Sodium Chloride and agar. Two methods of growing is usually practiced- the pour plate or the spread plate. The pour plate mixes the sample and the agar at 37°C and pours it onto a Petri dish.

Apparatus: -

NUTRIENT AGAR, 2 Petri-Dishes, flame, Incubator and oven

Procedure: - (Pour plate technique)

- Prepare the medium of (Total Plate Count) which written on the bottle
- Pour 1mL of the sample in one Petri-dish and 3mL in another Petri-dish
- Pour media onto every Petri-dish until covering it surface
- Put Petri-dish in the Incubator at 37°C for 48 hrs.
- After 48 hrs, check on the Petri-dish (Positive or Negative)

E-Coli and Coli-form

E-coli are a type of bacteria; that is, a fecal coli-form whereas the coli-form is a bacterium involved in the fermentation of lactose when incubated at 35–37°C. The other type of coli-form bacteria is non-fecal coli-forms that are Enterobacter and Klebsiella. Fecal coli-forms live inside the intestine of warm-blooded animals while non-fecal coli-forms live free in the soil.

a) **Coli-form** can be made Levine agar.

Typically this agar consists of Lactose, Eosin, Di-potassium hydrogen phosphate, Methylene blue and agar.

Apparatus: - (for Coli. form)

EOSIN METHYLENE BLUE AGAR, Petri-Dishes, flame, Incubator, oven, Buchner Filter, Filter paper and Vacuum

Procedure: - (Filtration Technique)

- Prepare the medium of (Coli-Form) which written on the bottle
- Pour medium on Petri-dish until coherent
- Filter 100mL of the sample in Buchner Filter by filter paper
- Pour these filter paper on Petri-dish
- Put Petri-dish in the Incubator at 37°C for 48 hrs.
- After 48 hrs, check on the Petri-dish (Positive or Negative)

b) E-Coli can be made MACCONKEY AGAR.

Typically this agar consists of Lactose, Peptone, Bile salts, Sodium Chloride, Neutral red and agar.

Apparatus: - (for E-Coli)

MACCONKEY AGAR, Petri-Dishes, flame, Incubator, oven, Buchner Filter, Filter paper and Vacuum

Procedure: - (Filtration Technique)

- Prepare the medium of (E-Coli) which written on the Bottle
- Pour medium on Petri-dish until coherent
- Filter 100mL of the sample in Buchner Filter by filter paper
- Pour these filter paper on Petri-dish
- Put Petri-dish in the Incubator at 37°C for 48 hrs.
- After 48 hrs, check on the Petri-dish (Positive or Negative)

Streptococcus [4]

KF Streptococcus agar; Typically this agar consists of Proteose peptone, Yeast extract, sodium chloride, Sodium glyerophosphate, Maltose, Lactose, Sodium azide, Bromocresol purple and agar

Apparatus: -
KF STREPTOCOCCUS AGAR, Petri-Dishes, flame, Incubator, oven, Buchner Filter, Filter paper and Vacuum pump

Procedure: - (Filtration Technique)

- Prepare the medium of (Streptococcus) which written on the bottle
- Pour medium on Petri-dish until coherent
- Filter 100ml of the sample in Buchner Filter by filter paper
- Pour these filter paper on Petri-dish
- Put Petri-dish in the Incubator at 35°C for 48 hrs.
- After 48 hrs, check on the Petri-dish (Positive or Negative)

Pseudomonas [5]

Pseudomonas agar base; typically this agar consists of Gelatin peptone, Casein hydrolysate, Potassium sulphate, Magnesium chloride and agar

Apparatus: -
Agar Base, 2 Petri-Dishes, flame, Incubator and oven

Procedure: - (CFC Technique)

- Firstly; prepare The (Agar Base) which written on the bottle
- Second; prepare (Pseudomonas CFC Medium)
- Pour medium on Petri-dish until coherent
- Pour 1 ml of the in one Petri-dish and 3 ml in another Petri-dish
- Put Petri-dish in the Incubator at 25°C for 48 hrs.
- After 48 hrs, check on the Petri-dish (Positive or Negative)

To prepare the Agar Base:-
Suspend (24.2 g) of the agar base, in (500mL) of distilled water. Add (5mL) of glycerol. Bring to the boil to dissolve completely, sterilize by autoclaving at 121°C for 15 minutes. Allow the medium to cool to 50°C.

To prepare Pseudomonas CFC Agar:-
To (500mL) of agar base cooled to 50°C add the contents of 1 vial of Pseudomonas CFC Supplement (SR0103) rehydrated as directed. Mix well and pour into sterile Petri dishes.

ANALYSIS FOR WASTEWATER: -

- **Dissolved Oxygen** [6]

The dissolved oxygen (DO) is oxygen that is dissolved in water
the oxygen dissolves by diffusion from the surrounding air; aeration of water and photosynthesis.

Photosynthesis (in the presence of light and chlorophyll):

$$Carbon\ dioxide + Water \rightarrow Oxygen + Carbon\ rich\ foods$$

$$\boldsymbol{6CO_2 + 6H_2O \rightarrow O_2 + C_6H_{12}O_6}$$

Dissolved Oxygen can be measure by titrimetric method or electrometric method

(1) **Titrimetric method** (Oxidizing property of DO)

There are different titrimetric methods based on the nature of sample to be tested

(a) Azide Modification

Interference caused by nitrate is removed effectively

(b) Alum Flocculation Modification

If the sample contains suspended solids

(c) Permanganate Modification

If the sample contains iron (Fe^{2+}) but this method is not useful when the sample contains sulphites, thiosulphates and high BOD.

(d) Winkler Method: -

$$MnSO_4 + 2KOH \rightarrow Mn(OH)_2 + K_2SO_4$$

$$2Mn(OH)_2 + O_2 \rightarrow 2MnO(OH)_2$$

$$MnO(OH)_2 + 2H_2SO_4 \rightarrow Mn(SO_4)_2 + 3H_2O$$

$$Mn(SO_4)_2 + 2KI \rightarrow MnSO_4 + K_2SO_4 + I_2$$

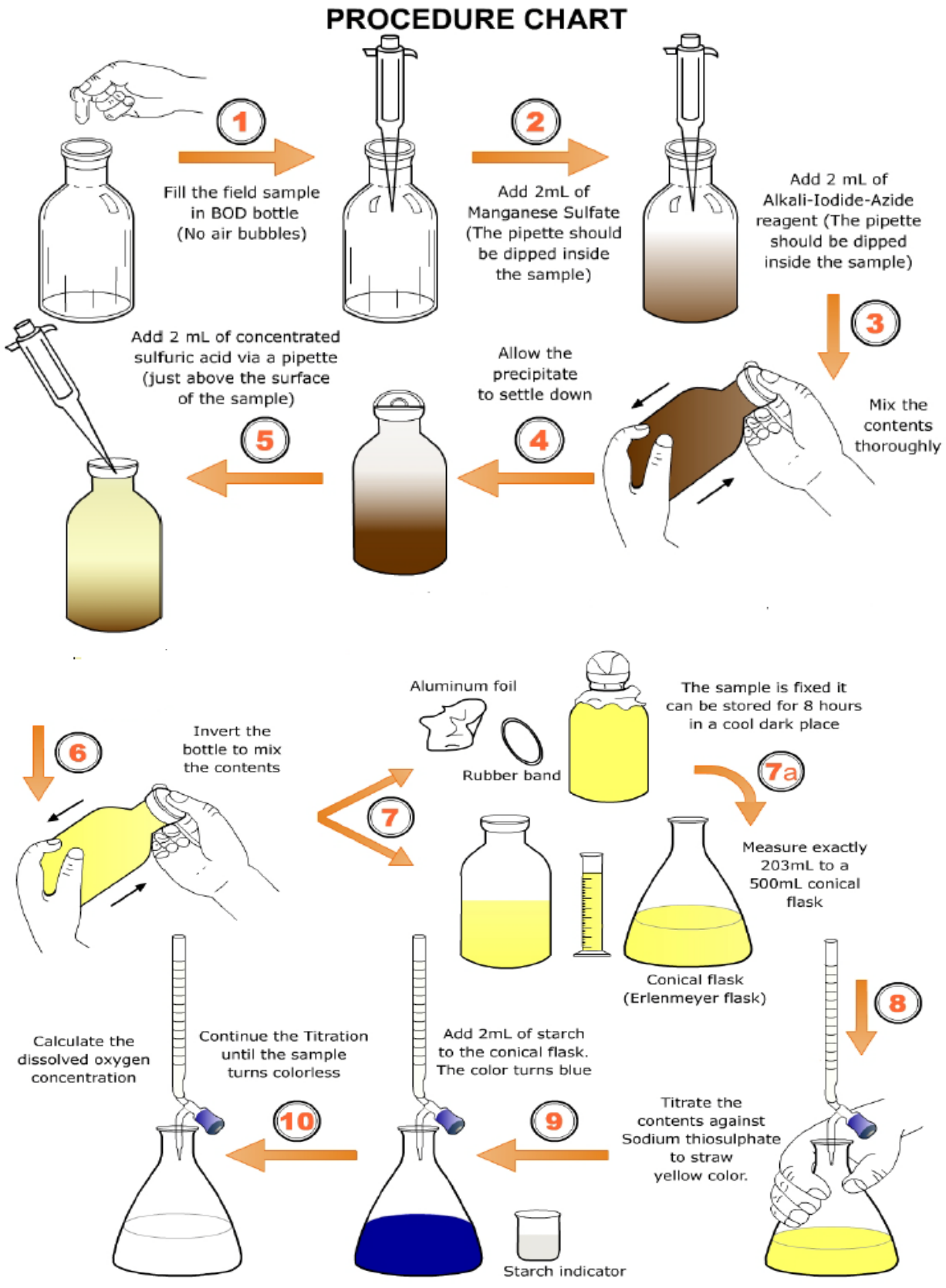
$$I_2 + 2{S_2O_3}^{2-} \rightarrow {S_4O_6}^{2-} + 2I^-$$

Procedure: -

- Take 300 ml of the sample Collected in BOD bottle
- Add 2 ml of Manganese sulfate to the BOD bottle

- Add 2 ml of alkali-iodide-azide reagent, if Oxygen is present, a brownish-orange cloud of precipitate or floc will appear
- Add 2 ml of Sulfuric acid (Conc.)
- Stored for up to 8hrs if kept in a cool, dark place
- Take 200 ml of the solution from the bottle
- Add (3 – 4) drop from Starch indicator solution, color become Blue
- Titrate against Sodium thiosulphate(0.025M) until Colorless
- Record the volume of Sodium thiosulphate (v)
- Calculated the Dissolving Oxygen from equation:-

$$DO = \frac{(v)_{Sodium\ Thiosulphate} * 0.2 * 1000}{(v)_{Sample\ slon.\ taken}}$$


PROCEDURE CHART
1
Fill the field sample in BOD bottle (No air bubbles)
2
Add 2mL of Manganese Sulfate (The pipette should be dipped inside the sample)
Add 2 mL of Alkali-Iodide-Azide reagent (The pipette should be dipped inside the sample)
3
Mix the contents thoroughly
Allow the precipitate to settle down
4
Add 2 mL of concentrated sulfuric acid via a pipette (just above the surface of the sample)
5
Aluminum foil
Rubber band
The sample is fixed it can be stored for 8 hours in a cool dark place
6
Invert the bottle to mix the contents
7
7a
Measure exactly 203mL to a 500mL conical flask
Conical flask (Erlenmeyer flask)
8
Titrate the contents against Sodium thiosulphate to straw yellow color.
9
Starch indicator
Add 2mL of starch to the conical flask. The color turns blue
10
Continue the Titration until the sample turns colorless
Calculate the dissolved oxygen concentration

Biochemical Oxygen Demand (BOD) [7]

BOD is the principle test to give an idea of the biodegradability of any sample and strength of the waste. Hence the amount of pollution can be easily measured by it.

Efficiency of any treatment plant can be judged by considering influent BOD and the effluent BOD and so also the organic loading on the unit.

BOD can be measured by Know DO

Firstly; we know prepare **dilution water** 3 to 5 days before initiating BOD test to ensure that the BOD of the dilution water is less than 0.2 mg/L: -

The required volume of water (five liters of organic free distilled water)
add 5 ml of Calcium chloride solution
add 5 ml of magnesium sulphate solution
add 5 ml of ferric chloride solution
add 5ml of phosphate buffer solution

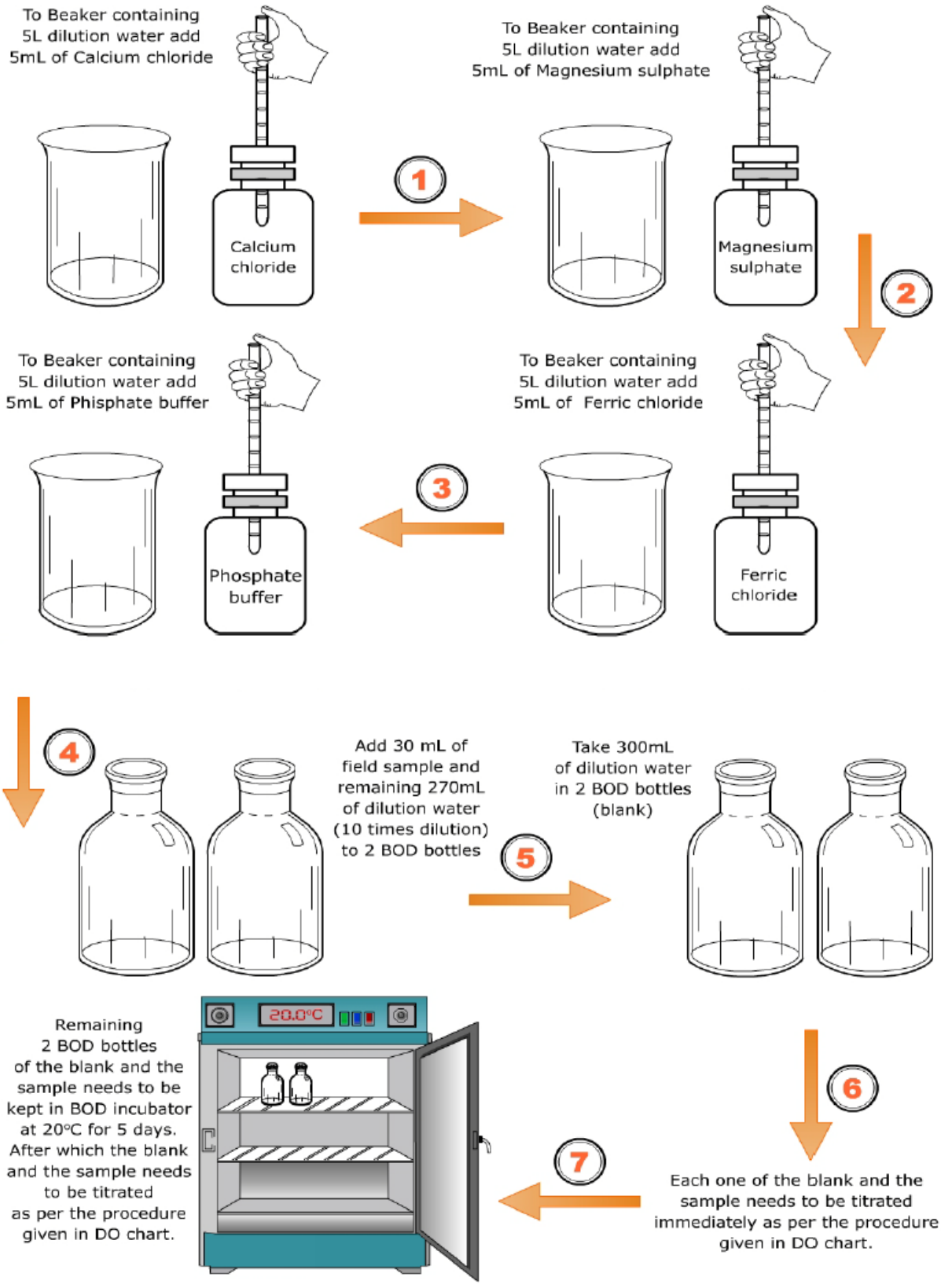
Produce: -

1- Take four BOD bottle Capacity 300mL (Two for blank and two for sample)
2- Add 10mL of the sample to each of the two BOD bottle and the fill the remaining quaintly with the dilution water (we diluted the sample 30 times)
3- The remaining the two bottles are for blank, to these bottles adds dilution water alone.
4- Now, preserve one bottle of blank and one bottle of sample in a BOD Incubator at 20℃ for 5 days… (DO_5)
5- The other two bottles (one for blank and one for sample) we use **Winkler Method** to calculate DO
6- Now, we can calculate the Chemical Oxygen Demand from equation: -

$$\boldsymbol{BOD = \frac{(DO - DO_5 - 0.2) * (v)_{diluted\ sample}}{(v)_{sample\ taken}}}$$

Which: -
DO = Dissolving Oxygen by Winkler method, $\boldsymbol{DO_5}$ = Dissolving Oxygen after 5 day
Value (0.2) = Blank for DO

PROCEDURE CHART



Chemical Oxygen Demand (COD) [8]

The chemical oxygen demand (COD) test is commonly used to indirectly measure the amount of organic compounds in water. Most applications of COD determine the amount of organic pollutants frond in surface water

COD is the measurement of the amount of oxygen in water consumed for chemical oxidation of pollutants.

COD determine the quantity of oxygen required to oxidize the organic matter in water or waste water sample, under specific conditions of oxidizing agent, temperature and time.

The ratio of BOD to COD greater than or equal to 0.8 indicates that waste water highly polluted and amenable to the biological treatment.

Procedure: -

- Take three COD vials with stopper (two for the sample and one for Blank)
- Add 2.5mL of the sample to COD vials and (2.5mL) distilled water to COD vials for Blank
- Add 1.5mL of potassium dichromate reagent for Three COD vials
- Add 3.5mL of sulphuric acid reagent
- Cap tubes tightly. Switch on the COD Digester and fix the temperature at 150℃ and set the time at 2 hrs.
- Transfer the contents of the sample COD vial to conical flask
- Add 1-2 drop of Ferroin indicator, with Blank Color change from bluish green to reddish brown and with sample color change from green to reddish brown
- Titrate against Ferrous ammonium sulphate (0.1 N)
- Calculate Chemical Oxygen Demand from equation:

$$COD = \frac{(A - B) * N * 8 * 1000}{(v)_{sample\ taken}}$$

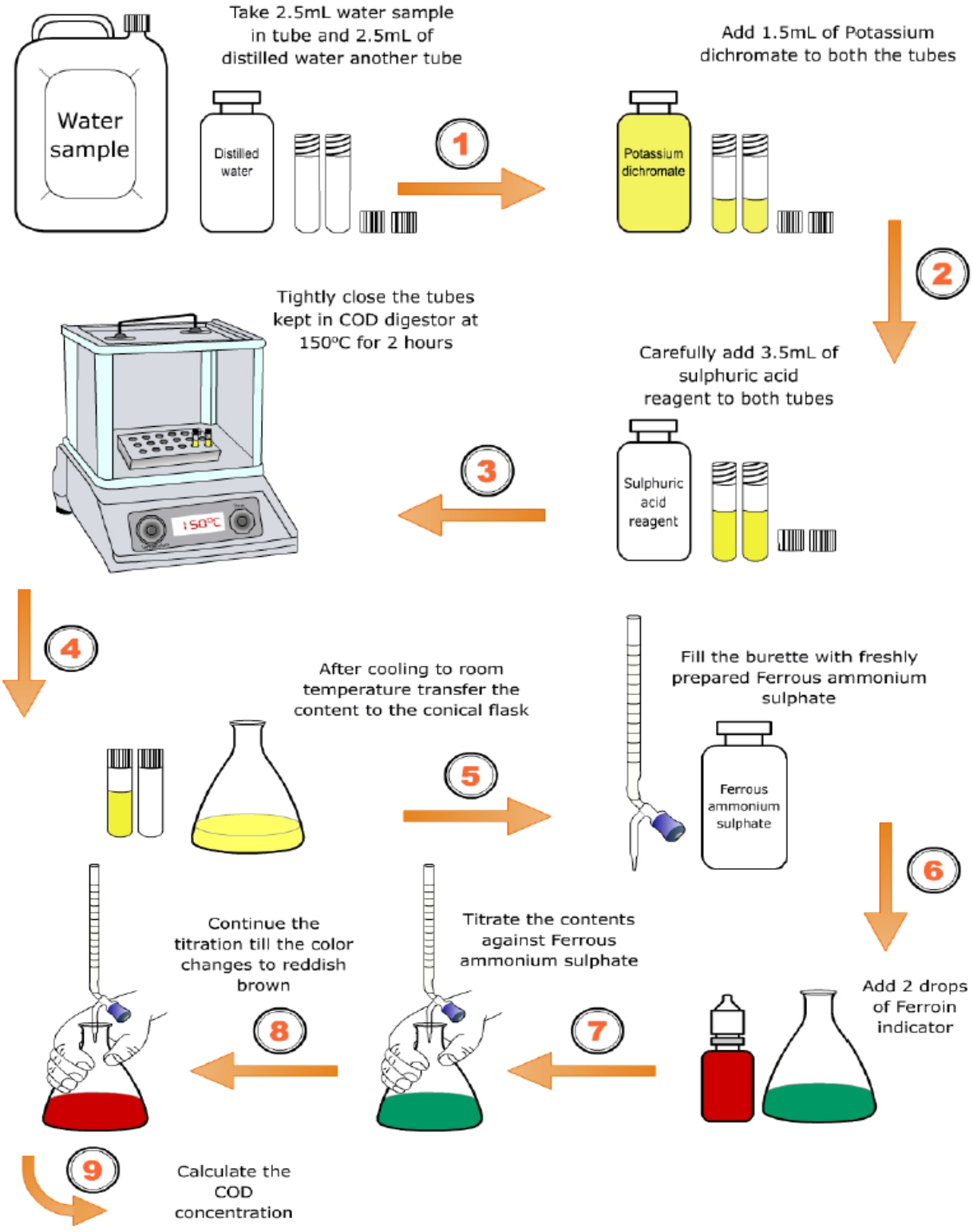
Where: -

A = Volume of Ferrous Ammonium Sulphate for blank

B = Volume of Ferrous Ammonium Sulphate for Sample

N = Normality of Ferrous Ammonium Sulphate

PROCEDURE CHART



Total Organic Carbon (TOC) [9]

Total organic carbon (TOC) is a measure of the carbon content of dissolved and un-dissolved organic matter present in the water. It does not give information on the nature of the organic substance.

Two types of carbon are present in water: -
Total Organic Carbon (TC) and Inorganic Carbon (TIC)

Definitions according to EN 1484

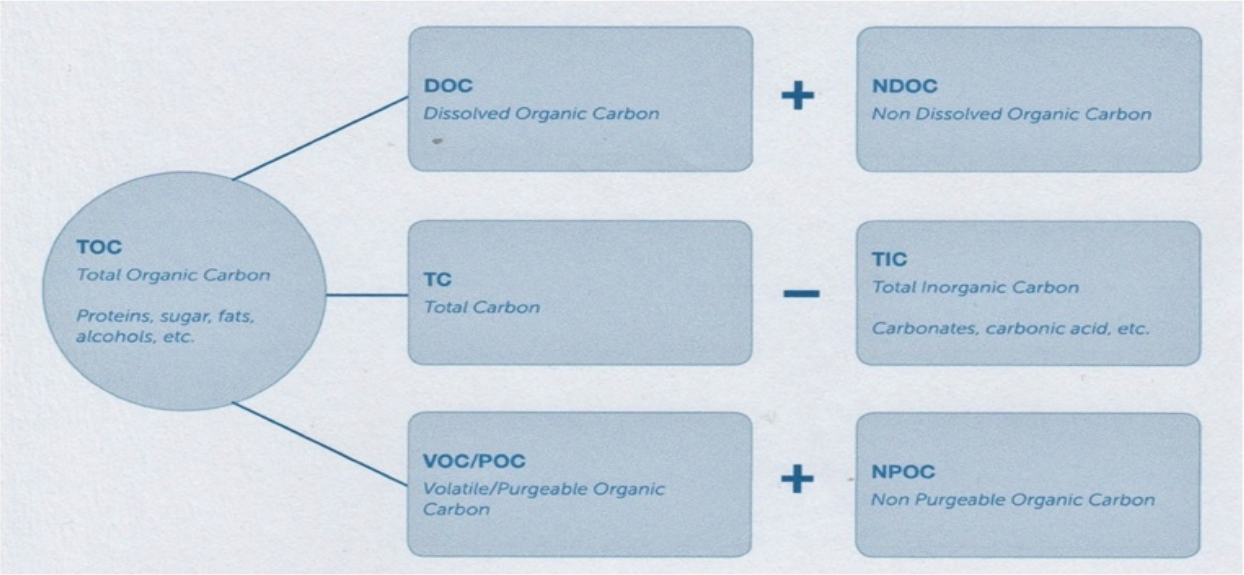
Total Carbon (TC): -
The sum of organically bound and inorganically bound carbon present in water, including elemental carbon

Total inorganic carbon (TIC): -
The sum of carbon present in water, consisting of elemental carbon, total carbon dioxide, carbon monoxide, Cyanate and thiocyanate. TOC instruments mostly register as TIC only the CO2 originating from hydrogen Carbonates and carbonates

Dissolved organic carbon (DOC): -
The sum of organically bound carbon present in water originating from compounds which will pass a membrane filter of pore size of (0.45µ) Cyanate and thiocyanate are also measured

Volatile organic carbon (VOC, POC): -
Under the conditions of this method purgeable organic matter (POC)

Non volatile organic carbon (NVOC, NPOC): -
Under the conditions of this method non-purgeable organic carbon (NPOC)



We can calculated TOC from the difference between (TC) and (TIC)

Determination of Total Carbon (TC): -

Can be measured by Oxidation and decomposition of the sample and detection of the deriving carbon dioxide CO_2 by using NDIR detection

Determination of Total Inorganic Carbon (TIC): -

Can be measured by degradation of the carbonates by acid, purging and detection of the deriving carbon dioxide CO_2 by using NDIR detection

$$\therefore TOC = TC - TIC$$

But disadvantaged this method (less suitable for high TIC concentrations)

- **Oil and Grease** [10]

Group of organic substance soluble in an organic solvent
Oil: - water insoluble organic material (Liquid at room temperature)
Grease: - water insoluble organic material (Solid or semi-solid at room temperature)
Extractable materials: - non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases, and related materials

We have three methods to determine Oil and Grease in water and wastewater as follow

(a) **Soxhlet extraction method** from (20 – 200 mg/L)

(b) **Infra-red method** from (0.5 - 100 mg/L)

Extracted hydrocarbons absorb IR energy at a common IR wavelength, and amount of energy absorbed is proportional to concentration of OG in solvent.

(c) **Gravimetric method** is applicable to the estimation of O&G from (4 - 100 mg/L)

Apparatus: -

Separating Funnel (1 Liter Capacity with Teflon)
water bath, distillation flask
whatman40 filter paper, 11cm, or equivalent

Reagent: -

Hydrochloric acid (1:1)
1, 1, 2-Tri-chloro-1, 1, 2- Tri-fluoroethane
Sodium Sulphate (anhydrous)

Produce: -

- Transfer the acidified sample (we make sample have pH=2 by add concentrated Hydrochloric acid) to Separating Funnel with (30mL) of Tri
- Add the solvent washings to the Separating Funnel
- Shake vigorously for about 2 minutes
- If it is suspected that a stable emulsion will form, shake gently for 5 to 10 minutes
- Let the layer separated
- Drain the solvent layer through a funnel containing moistened filter paper into a clean distillation flask
- If a clear solvent layer cannot be obtained add (1.0 g) of Sodium Sulphate to the filter paper
- Extract two more times with 30ml of solvent each time, but first rinse the sample container with the solvent
- Collect the extracts in a distillation flask and wash filter paper with an additional 10 to 20 ml of the solvent
- Distill the solvent from distillation flask over a water bath at 70℃
- Place the beaker on water bath for 15 minutes at 70℃ and evaporate off all solvents
- Cool the beaker for 30 minutes and weigh
- Calculated Oil and Grease from equation: -

$$\therefore Oil\ and\ Grease = \frac{M}{V} * 1000$$

Where: -

M = mass of the residue, **V =** volume of the sample taken

PREPARATION OF REAGENTS AND SOLUTIONS: -

- **Phenol Red indicator: - for (pH)**

Dissolving (0.1 g) of phenol red in (2.82mL) of 0.1M NaOH and add (20mL) of ethanol (95%) and complete volume to (100mL) of distilled water

- **Potassium Chromate indicator: - for (Chloride)**

Dissolving (1.0 g) of potassium chromate in (20mL) of distilled water

- **Methyl Orange indicator: - for (Total Alkalinity)**

Dissolving (0.1 g) of methyl orange in (80mL) of water and complete volume to (100mL) of ethanol (95%)

- **Phenolphthalein indicator: - for (P-Alkalinity)**

Dissolving (0.1 g) of Phenolphthalein in (80mL) of ethanol (95%) and complete volume to (100mL) of distilled water

- **Eriochrome Black T indicator mixture: - for (Total Hardness)**

Mixing (1.0 g) Eriochrome Black T with (100.0 g) Sodium Chloride and grinding this mixture

- **Buffer Solution pH= 10 : - for (Total Hardness)**

Dissolving (16.9 g) of ammonium chloride in (143mL) of concentrated ammonium hydroxide and then diluted to (250mL) with distilled water

- **Muroxide indicator mixture: - for (Calcium Hardness)**

Mixing (0.2 g) Muroxide with (100.0 g) Sodium Chloride and grinding this mixture

- **Manganese sulphate Solution: - for (Dissolved Oxygen)**

Dissolving (50.0 g) of manganese sulphate in (100mL) of distilled water

- **Alkali-iodide-azide Solution: - for (Dissolved Oxygen)**

Dissolving (50.0 g) of sodium hydroxide and (15.0 g) of potassium iodide in (100mL) of distilled water

- **Starch Indicator: - for (Dissolved Oxygen)**

Dissolving (2.0 g) of starch powder in (100mL) of hot distilled water

- **Sodium Thiosulphate Solution: - for (Dissolved Oxygen)**

Dissolving (3.1025 g) of sodium thiosulphate and (0.4 g) of sodium hydroxide in (1000mL) of distilled water

- **Calcium Chloride Solution: - for (Biochemical Oxygen Demand)**

Dissolving (2.75 g) of calcium chloride in (100mL) of distilled water

- **Ferric Chloride Solution: - for (Biochemical Oxygen Demand)**

Dissolving (0.25 g) of Ferric chloride in (1000mL) distilled water

- **Phosphate Buffer Solution: - for (Biochemical Oxygen Demand)**

Dissolving (0.85 g) of potassium dihydrogen phosphate, (2.18 g) of dipotassium monohydrogen phosphate, (3.34 g) of disodium hydrogen phosphate and (0.17 g) of ammonium chloride in (70mL) distilled water and complete to (100mL), the pH of the solution will be 7.2

- **Potassium dichromate Reagent-Digestion Solution: - for (Chemical Oxygen Demand)**

Dissolving (4.913 g) of potassium dichromate, (33.3 g) of mercuric sulphate, (167mL) of Sulphuric acid (Conc.) and complete volume to (1000mL) of distilled water

- **Sulphuric acid reagent-Catalyst Solution: - for (Chemical Oxygen Demand)**

Dissolving (5.5 g) of Silver sulphate crystals in (500mL) of Concentrated Sulphuric acid (stay this step for 24hrs until silver sulphate dissolve completely)

- **Ferrous Ammonium Sulphate Solution: - for (Chemical Oxygen Demand)**

Dissolving (39.2 g) of ferrous ammonium sulphate in (1000mL) of distilled water

- **Ferroin Indicator: - for (Chemical Oxygen Demand)**

Dissolving (1.48 g) of 1, 10-phenanthroline monohydrate, (0.7 g) of ferrous sulphate in (100mL) of distilled water

- **Preparation method of (NaCl, $AgNO_3$, H_2SO_4, NaOH, EDTA)**

Molarity (M): - is the ratio of moles to volume of the solution (mol/L) or is the number of moles of a substance per liter of solution.

In case of Solid Substance:-

$$\therefore gm = M * M.wt * V(L)$$

Which: -
gm = the weight of Substance in grams, **M =** Molarity required
M.wt = Molecular weight of Substance, V **(L) =** Volume per Liter

In case of Liquid Substance:-

$$\therefore V = \frac{M.wt * M * v\,(L) * 100}{D * c}$$

Which: -

V = the Volume of Liquid Substance, **M =** Molarity required

M.wt = Molecular weight of Substance, **v (L) =** Volume per Liter

D = Density of Liquid substance, **C =** Concentration of Liquid substance.

Normality (N): - is defined as the number of mole equivalents per liter of solution.

In case of Solid Substance: -

$$\therefore gm = \frac{N * eq.wt * V\,(L)}{1000}$$

In case of Liquid Substance: -

$$\therefore Normality = \frac{C * D * 10}{eq.wt}$$

Which: -

D = Density of Liquid substance, **C =** Concentration of Liquid substance

- **How to Calculate Equivalent weight?**

In Case of Acid

$$eq.wt = \frac{M.wt\ of\ substance}{number\ of\ Hydrogen\ replaced}$$

In case of Basic

$$eq.wt = \frac{M.wt\ of\ substance}{number\ of\ Hydroxide\ replaced}$$

In case of Salt

$$eq.wt = \frac{M.wt\ of\ slat}{valence\ one\ of\ salt * Number\ of\ repetitions}$$

Then we used this equation

$$\therefore (N * V)_{befor} = (N * V)_{after}$$

How to calculate Normality from Molarity?

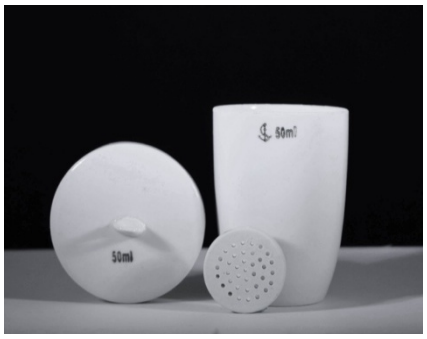
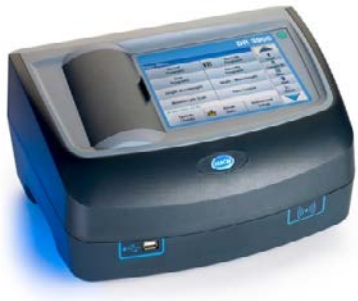
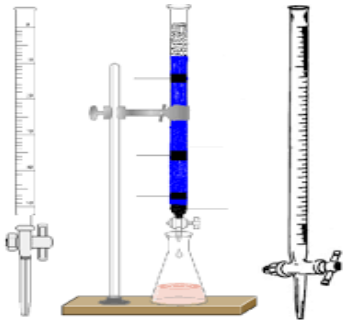
The mole equivalents of an acid or base are calculated by determining the number of H^+ or OH^- ions per molecule: -

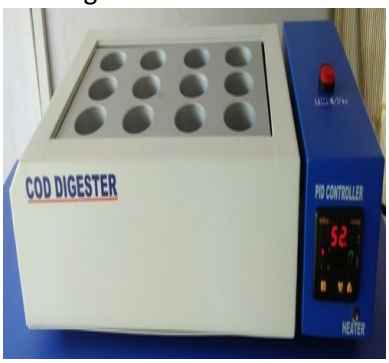
$$\therefore N = n * M$$

Where: -

n = is an integer

TOOLS USED IN EXPERIMENTS: -

Myron (4P , 6P) 	HANNA 	pH meter 
Gooch crucible 	Vacuum filter 	Filter paper 
Phenol red 	Conical Flask 	Cuvette 10ml 
DR3900 	burette 	Nessler tubes 

Bottle glass 	Petri-Dishes 	Incubator 
Oven 	NUTRIENT AGAR 	Levine agar 
Pseudomonas agar base 	MACCONKEY AGAR 	BOD bottle 
COD Vials 	COD Digester 	Separating Funnel 

whatman40 filter paper	distillation flask	Water bath
		

REFERENCE: -

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[6] IS: 3025 (Part 38) – Reaffirmed 2003

[7] IS: 3025 (Part 44) – Reaffirmed 2003

[8] IS: 3025 (Part 58) – Reaffirmed 2003

[9] EN1484_European Standard Guidelines for TOC

[10] IS: 3025 (Part 39) – Reaffirmed 2003 & weftec 2010 www.weftec.org