GOVT CO-ED POLYTECHNIC

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LAB MANUAL

Branch : For Electrical & Civil Engineering

Year & Semester : 1st Year / 2nd Semester

2000291(011) – Applied Chemistry (Lab)

General Chemistry Safety and Laboratory Rules

1. DO NOT perform unauthorized experiments or work in a laboratory alone.
2. Approved eye protection must be worn at all times in the laboratory. Tennessee State law requires the use of such devices. Eye protection must be splash proof chemical goggles and be approved by your instructor. If you do get a chemical in your eye rinse immediately with large quantities of water using the eye-wash stations.
3. Long hair and loose clothing must be confined while in a laboratory.
4. Appropriate clothing must be worn at all times while in the laboratory. Your legs must be completely covered below the knee by your choice of clothing. If your clothing does not meet the requirement you may choose to wear an approved laboratory coat or apron which does cover your legs to your knees.
5. Closed shoes with socks must be worn at ALL times – open-toed shoes, backless shoes, sling backs, clogs, and sandals are not permitted
6. Know the location and proper use of fire extinguishers, fire blankets, safety showers, eye wash devices and first aid kits.
7. Before obtaining any chemicals carefully read the label on the reagent bottles.
8. Thoroughly wash your hands after leaving the laboratory.
9. Eating, smoking, and drinking are not allowed in a chemistry laboratory.
10. Use the fume hoods when toxic or irritating vapors are involved.
11. Mouth suction is never used to fill a pipette.
12. Never direct the open end of test tube toward yourself or anyone else.
13. Never pour water into concentrated acid.
14. Learn the proper procedure for igniting and operating a laboratory burner. Always extinguish the flame when the burner is not being used. Make sure that all flammable reagents are well removed before.
15. Liquid and solid waste containers must be properly used at all times.
16. Never place chemicals directly on the balance pan. Always use a proper weighing container when using a balance to weigh a chemical. Never pour chemicals directly over the balance.
17. Never return unused chemicals to their original container (unless directed to do so by the instructor).
18. Securely replace lids, caps, and stoppers after removing reagents from containers.
19. Always wipe spatulas clean before and after inserting into reagent bottles.
20. Report any accident and/or injury, however minor, to your instructor immediately.

Best Wishes for this academic session
Applied Chemistry
Government Polytechnic Raipur

List of Experiments

S. No.	**Name of Experiments**
1.	To estimate the amount of total hardness present in the given sample of water by EDTA titration method.
2.	To determine the relative viscosity of the given liquid at room temperature
3.	Determination of the calorific value of a given sample of fuel (liquid and solid) by bomb calorimeter.
4.	To determine the Flash Point and Fire Point of lubricant by Pensky Martin apparatus.
5.	To measure the pH of different solution
6.	Proximate analysis of a sample of Coal.
7.	To determine the alkalinity of given sample of water using N/100 hydrochloric acid solution and methyl orange and phenolphthalein as indicators.
8.	To determine the amount of dissolved oxygen present in a given water sample .
9	To standarization of KM_nO_4 solution using standard oxalic acid solution
10	To determine the ferrous iron present in Mohrs salt by using standard KM_nO_4 solution

ESTIMATION OF HARDNESS OF WATER BY EDTA METHOD

Experiment No. ……1……….   Date:…………………

Aim : **To estimate the amount of total hardness present in the given sample of water by EDTA titration method.**

Apparatus required :

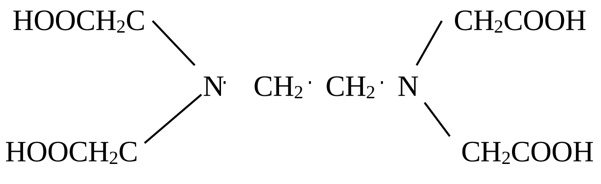
1. 50 ml Burette
2. 20 ml Pipette
3. 250 ml Conical flask
4. 100 ml Beaker
5. 250 ml
6. beaker
7. Glass funnel

Reagents :

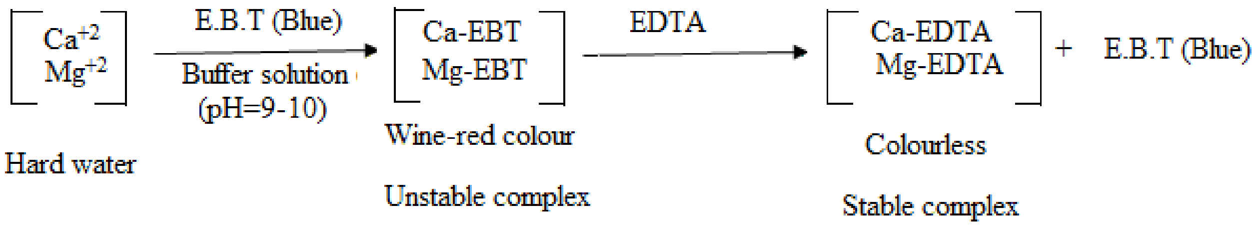
1. EDTA solution (Ethylene diamine tetra acetic acid
2. Standard $CaCO_3$ solution
3. Eriochrome Black–T indicator
4. Buffer solution (pH - 10)

Theory : EDTA Ethylenediamine tetra acetic acid) forms colorless stable complexes with Ca^{2+} and Mg^{2+} ions present in water at pH = 9-10. To maintain the pH of the solution at 9-10, buffer solution (NH_4Cl + NH_4OH) is used. Eriochrome Black-T (E.B.T) is used as an indicator.

The sample of hard water must be treated with buffer solution and EBT indicator which forms unstable, wine-red colored complex s with Ca^{2+} and Mg^{2+} present in water



Structure of EDTA



Procedure : ***1. Standardization of EDTA***

(i) Pipette out 20 ml of standard hard water into a conical flask.

(ii) Add 5 ml of buffer solution and few drops of Eriochrome Black-T. The indicator, which is originally blue color would acquire a wine-red color.

(iii)Titrate with EDTA solution taken in the burette, till the wine red color changes to blue which is the end point. Let the burette reading of EDTA be V ml.

2. Determination of Total hardness

Repeat the above titration method for sample hard water instead of standard hard water. Let the burette reading of EDTA be V ml

3.Determination of Permanent hardness

Take 100 ml of sample hard water in 250 ml beaker. Boil it to remove temporary hardness to about half of this volume and cool to room temperature Filter through filter paper to remove insoluble $CaCO_3$ and $MgCO_3$. Make up the volume to the original 100 ml by adding distilled water. Now pipette out 20 ml of this solution into a clean conical flask. Then repeat the process of titration steps as mentioned above. Let the burette reading of EDTA be V ml.

Observations : ***1. Standardization of EDTA***

Titration of Known water sample with EDTA solution

S.No.	Volume of water sample taken (ml)	Burette reading		Concordant Volume of M/100 EDTA solution used (ml)
		Initial	Final	
1				
2				
3				

2. Determination of Total hardness

Titration of given water sample with EDTA solution

S.No.	Volume of water sample taken (ml)	Burette reading		Concordant Volume of M/100 EDTA solution used (ml)
		Initial	Final	
1				
2				
3				

Calculations: ***1. Standardization of EDTA***

$$M_1V_1 = M_2V_2$$

Where, M_1= Molarity of standard hard water

V_1= Volume of standard hard water in conical flask

M_2= Molarity of EDTA

V_2 = Volume of EDTA consumed (burette reading)

2.Determination of Total hardness

$$M_3V_3 = M_2V_2$$

Where, M_3= Molarity of unknown hard water

V_3= Volume of unknown hard water in conical flask

M_2= Molarity of EDTA

V_2 = Volume of EDTA consumed (burette reading)

Note multiply M_3 with 10^5 to covert hardness into parts per million (ppm).

Strength (total hardness) = Normality X Equivalent weight of CaCo$_3$
in termsof ppm

Result : The hardness of the given water sample …………………………ppm.

Precautions : 1 All the volumetric apparatus should be washed with distilled water and dried before used.

2. Rinse the burette and pipette with a solution to be taken in it.

3. Maintain the pH at 8-10 during titration.

4. Use disodium salt of EDTA.

Viva-voce : 1. What do you mean by hardness of water?

2. What is EDTA? Gives its structure.

3. What do you mean by complexometric titration?

4. What are the types of hardness in water?

5. What is the unit of hardness?

Determination of viscosity through Redwood viscometer

Experiment No. …2…………. Date:…………………

Aim : **To determine the viscosity of lubricant oil by means of Redwood Viscometer.**

Apparatus required :

1. Redwood viscometer
2. Stop watch.

Reagents : Lubricating Oil

Theory : viscosity is a property of a liquid by virtue of which it offers resistance to its own flow . Coefficient of viscosity is defined as the force in dynes required to maintain unit relative velocity (1 cm/sec.) between two layers with unit distance (1cm) apart. The unit of viscosity is poise or centipoises.

In redwood viscometer , viscosity is measured as the time in seconds required for 50 ml of the lubricant to flow through a standard orifice under a def inite set of conditions and is expressed in redwood seconds.

Redwood Viscometer : Redwood viscometer consist of a cylindrical brass oil cup the holds the given lubricating oil. At the lower end , oil cup has been provided with a tape- ring central hole in which a jet is fixed. A small ball resting on the jet serves as the valve . The oil cup is surrounded by water bath for adjusting as the tempe rature . A special kind of narrow necked flask called Kohlrausch flask having a capacity of 50 ml is used for receiving the oil below the jet

Procedure :

1.Level the viscometer with the help of levelling s

2.Close the jet or orifice of the oilcup with the help of bob.

3.Fill the oil cup with the given lubricating oil upto the pointer. Fix one thermometer the given lubricating oil in the oil cup.

4.Fill the water in in the heating bath and fix another thermometer in it.

5.Start heating the side tube till the desired temperature of the oil is reached.

6.place the kholrausch flask below the jet. Lift the brass ball and start the stop watch.

7. Note the time taken for 100 ml of oil to go through the orifice.

8. Repeat the experiment 3-4 times and find the mean of the time taken in

second

Result : The relative viscosity of the given liquid with respect to water at room temperature is _______

Precautions :

1. The viscometer should be thoroughly cleaned.
2. Viscometer must be strictly kept in vertical position
3. Same volumes of liquid and water are to be taken while performing the experiment

Viva-voce :

1. What do you mean by viscosity
2. What is relative viscosity? Gives its formula..
3. What do you mean by density?
4. What is the unit of viscosity?
5. What are lubricants

Calorific value determination by using Bomb Calorimeter

Experiment No. …3…………. Date:…………………

Aim	:	**Determination of the calorific value of a given sample of fuel (liquid and solid) by bomb calorimeter.**
Apparatus required	:	Bomb calorimeter
Reagents	:	fuel sample
Theory	:	Calorific value of any fuel tells about its heating value which is the most important property of a fuel. Thus to know about a fuel it is necessary to know its Calorific value. Calorific value of a fuel is defined as the quality of heat energy given out upon the complete combustion of a unit mass of the fuel. The heat is commonly expressed either in the British thermal unit (Btu) or in metric system as Calorie.

Grass or higher calorific value

This is defined as the total amount of heat liberated by complete combustion of unit mass of fuel and by cooling down the end products of combustion to room temperature.

Net or lower calorific value

It is the calorific value determined the Net heat produced by complete combustion of unit mass of fuel and end products of combustion are allowed to be escaped in the atmosphere.

Relation between Gross and Net calorific values:

Net calorific value = Gross C.V. – Latent heat of condensation of water vapor Produced.

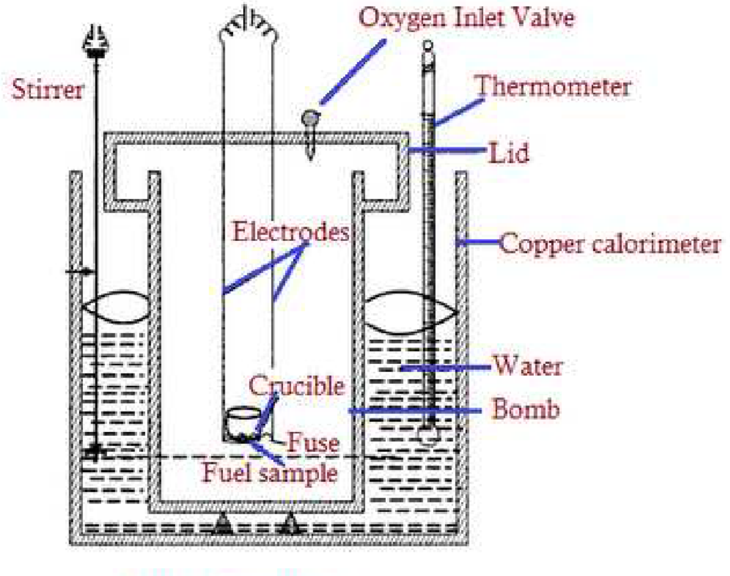
G C V – mass of hydrogen per unit weight fuel burnt X 9X Latent heat of condensation of water vapor

Where NCV = G CV –94/100 x 587cals/g
G C V –0.09 x 587cals/g

H = % of H2 in the fuel

Later heart of steam = The fuel 587cals/g

Diagram :



Bomb Calorimeter

Description of apparatus : **1. *Combustion bomb:*** This is a cylindrical vessel of ml capacity made of strong acid and alkali proof steel. The inner fixtures of the bomb are similarly made of heat and acid resistant stainless steel. It is equipped with a cover, which has provisions for inlet and outlet of oxygen, electrical connections and bearing the crucible containing the sample

2. *Calorimeter vessel:* The bomb assembly (A) is placed in the calorimeter vessel (B) which is cylindrical in form and made of brightly electroplated and polished brass-sheet having a capacity of about 3-5 litres. This contains a known volume of water.

3. Calorimeter jacket or thermostat: The calorimeter vessel along with the bomb is placed in a double walled asbestos-insulated cylindrical vessel. The space between the walls is filled with water at a temperature 2-30C below the room temperature. The cover of the thermostat is made of heat insulating material and posses holes for insertion of electric wire, thermometer (T) and stirrer (S).

4. Thermometer: The temperature of the calorimeter water is recorded with a Beckmann,s thermometer which can read accurately one hundredths part of a degree.

5. Stirrer: The water of the calorimeter is stirred with the help of an electric motor driven stirrer (S) which is operated with the help of a transformer.

Procedure **:** 1. Accurately weighed quantity of the given fuel is taken in a crucible fuel is crushed and compressed in the form of a pellet. Accurately weighed piece of fuse wire is stretched between the electrodes, touching to fuel pellet and oxygen is filled in the bomb at 25 atmosphere pressure.

2. Weighed quantity of water filled in the copper calorimeter. Beckmans thermometer and stirrer are fitted in it. Initial temperature is recorded.

3. Bomb is kept inside the calorimeter. The stirrer is worked and initial temperature of water is noted

4. Electrodes are connected to the battery. Fuse wire catches fire and initiates the combustion of fuel pellet. The heat produced due to the combustion of fuel pellet is transmitted to the water in the copper calorimeter.

5. After half an hour the Bomb is taken out and washed with distilled water. Bomb washing is made upto a definite volume to determine the H2S04 and HNO3 in it.

: Water equivalent of calorimeter is defined as the number of calories required to heat the calorimeter by 1°c

$w = M S$

M = Mass of calorimeter

S = Specific Heat

$W = M_1 S_1 + M_2 S_2 + M_3 S_3$

Observation : Calorific value of fuel = Q

Weight of fuel = x gm

Weight of water in copper calorimeter = W g

Water equivalent of calorimeter = w g

[Water equivalent of calorimeter is determined by burning a fuel of known calorific value] .

Initial temperature of water = T_1°c

Final temperature of water = T_2°c

Calculation : **According to Principle of Calrionimetry**

Heart Liberated=Heart Gained

Heat Gained by water = W x Δt x specific heat of water

= W (T2 – T1) cal.

Heat Gained by calorimeter = w (T2 – T1) cal.

Heat Liberated by fuel = Heat gained by water and calorimeter

mQ = (W + w) (T2 – T1)

Q = (W + w) (T2 – T1)/m

Result : The gross calorific value of the given fuel sample by bomb calorimeter is found to be ....calories/g.

Precautions : 1. Tighten the lid properly.

2. Check the working of stirrer.

3. Temperature should be recorded properly.

Viva-voce : 1. What is surface tension?

2. Write the unit of surface tension.

3. Why drops of water is spherical.

Determination of Flash Point and Fire Point of lubricant by Pensky Martin apparatus.

Experiment No. ………4…….   Date:…………………

Aim : **To determine the Flash Point and Fire Point of lubricant by Pensky Martin apparatus.**

Apparatus required :

1. Pensky Marten’s flash and fire point apparatus,
2. match box
3. thermometer
4. filter paper.

Reagents : Given lubricating oil, suitable organic solvent like CCl4, ether, petroleum, spirit or benzene

Theory : **Principle :**

Flash point is the lowest temperature at which the lubricating oil gives off enough vapors that ignite for a moment when tiny flame is brought near it.

Fire point is the lowest temperature at which the vapors of the oil burn continuously for at least five seconds when a tiny flame is brought near it.

Significance

Flash and fire points are used to indicate fire hazard of petroleum products and evaporation loses under high temperature. This test is of immense importance for illuminating and lubricating oils. It givesus the idea about the maximum temperature below which the oil can be used. It is used as the means of identification of specific lubricating oil for detection of contamination in the given lubricating oil

Description of Pensky Marten’s apparatus

It is used to determine the flash point of the lubricating oils, fuel oils, solvents, solvent containing material and suspension of solids.

This apparatus is used for the oil flashing between 490C and 3700C.

A Pensky Martin apparatus consists of the following major parts:

Oil cup (A): It is a cylindrical vessel, made of brass, with a filling mark grooved inside near the top. The inside of the cup is of slightly larger diameter above the filing mark and is covered with a lid.

Lid (B): It is equipped with the following parts:

(I) Stirrer: The stirring device consists of a vertical steel shaft (C) mounted in the centre of the cup and carrying two two-bladed brass propellers.

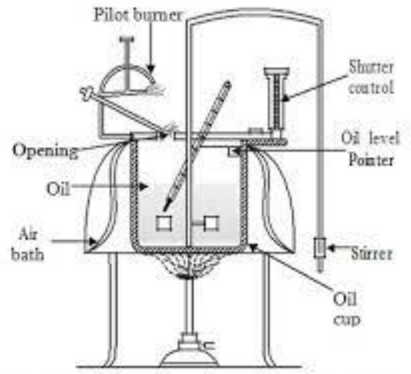


Fig. Pensky Marten's flash point apparatus

(II) Cover proper: It is made up of brass having a rime projecting downward and fitting the outside of the cup closely. It has four openings: one for thermometer (T); and the rest for the oxygen entry and exposure of vapors to the test flame.

(III) Shutter: The lid is equipped with a brass shutter (D) operating on the plane of the upper surface of the cover proper. The shutter is so shaped and mounted on the lid that when in one position, the holes are completely closed when in the other, these orifices are completely opened.

(IV) The flame exposure device: The lid is equipped with a pilot-lamp (L) with such a mechanism that its flame operates simultaneously with the shutter. When the shutter is in the 'open' position, the tip is lowered down in the centre of the central orifice.

Stove: The cup is heated by means of a properly stove (C) which consists of an 'air bath' (E) and a top plate (F). The air-bath has cylindrical interior about 4 cm deep and can be heated by a direct flame or an electric resistance element. The top-plate is also made of metal and mounted with an air gap between it and air bath.

Procedure :

1. Clean and dry all parts of the apparatus with the help of suitable solvent e.g. CCl_4, ether, petroleum spirit or benzene and dry it to remove any traces of solvent.

2. Fill the oil cup with the test oil up to the mark.

3. Fix the lids on the top through which are inserted a thermometer and a stirrer. Ensure that the flame exposure device is fixed on the top.

4. Light the test flame and adjust it to about 4 mm in diameter.

5. Heat apparatus as temp. of oil increases by 5 to 60 per min. as stirrer is continuously rotated at rate 1 to 2 rps.

6. At every 1^0 C rise of temp. Introduce test flame into the oil vapor. This is done by operating the shutter. On moving knob of shutter, test flame is lowered in oil vapors through
7. When test flame causes a distinct flame in interior cup, note temp. which represent the flash point.

8. Further heat the oil at the rate of 10C/ min. and continue applying the test flame as before.

9. The temperature at which the vapors of the oil give a clear and distinct blue flash for five seconds is recorded as the fire point of the oil.

Result :

1. The flash point of given oil sample=___ ^{0}C.

2. The fire point of given oil sample=____ ^{0}C.

Precautions :

1. The apparatus should be thoroughly dried. There should be no trace of moisture inside the cup.

2. The thermometer bulb should dip into the oil.

3. While applying the test flame, stirring should be continued.

4. Fill the sample of the lubricating oil up to the mark. There should be no oil on the outer part of the cup.

5. Avoid breathing over the surface of the oil.

Viva – voce :

1. What is flash point?
2. What is fire point?
3. What is lubricant?
4. Explain mechanism of lubrication.
5. Explain types of lubricants.

To measure the pH of different solution.

Experiment No. ……5………. Date:…………………

Aim : **To measure the pH of different solution**

Apparatus required : 1.Digital pH meter

2.Beaker

3.Buffer solution

Principle : Hydrogen ion concentration of a solution is a very important quantity in analytical chemistry. As a matter of fact it is difficult to express pH value in words and Sorensen‟s method is a mathematical one.

Theory : This is represented as

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

Or

$$pH = \log_{10} 1 - \log_{10} [H^+]$$

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

When the pH value of a solution is from Zero to less than seven then the solution will be acidic and when it is from above 7 to 14, then it is alkaline. At pH =7 the solution will be neutral.

Method : Connecting the electrode:

a) Set up the electrode stand and fit the pH electrode into it.

b) Carefully remove the procreative rubber cap from the filling hole of electrode. The level of KCl solution should be a few mm below the hole. Top up if necessary with saturated KCl solution. Now put the rubber cap back.

2) Preparation of buffer solutions: Dissolve one buffer tablet or powder pack of 7 pH in 100 ml distilled water. The pH of this solution is 7.

3) Calibration of Electrode:- The electrode should be calibrated before beginning measurements.

Following procedure is adopted for calibration of electrode.

a) Connect the combination pH electrode to the input socket, wash it with water and switch on the instrument.

b) Dip the electrode in 7 pH buffer solution.

c) Set the "TEMPERATURE OC" control to the buffer solution temperature.

d) Set the function selector switch to „pH‟ position and adjust with "CALIBRATE" control till the digital and display shows the precise pH value of the buffer solution.

e) Now move the function selector switch to „STAND BY‟.

f) Remove the electrode from the buffer solution and wash it with distilled on de-iodised water.

g) Dip the combination electrode into another buffer solution (4pH).

h) Set the "TEMPERATURE OC" control to the temperature of the selected buffer solution.

i) Set the function selector switch to pH position adjusts the „SLOPE‟ correction control, at the front panel until the display shows the pH value of the selected buffer solution. Check that the correct readings are obtained with both the buffer solutions without further adjustment

Observation Table : **pH of different given sample**

S. No.	**Solutions**	pH value
1	**Solution No. 1**	**4.5**
2	**Solution No. 2**	**5**
3	**Solution No. 3**	**6**
4	**Unknown**	

Result :

pH of Solution No. 1 =

2) pH of Solution No. 2 =

3) pH of Solution No.3 =

4) pH of unknown water sample -------

Precautions : 1) The electrode should be calibrated before beginning measurements.

2) Buffer solution should be stored in a cool place

Viva – voce :

1. What is pH?
2. What is buffer solution?
3. What is SHE?
4. What is pH of SHE..
5. Explain types of buffer solution.

Proximate analysis of a sample of Coal.

Experiment No. ………6…….   Date:…………………

Aim : **Proximate analysis of a sample of Coal.**

Apparatus required :

1. Muffle furnace
2. Desiccators
3. Oven
4. Crucible
5. Bunsen Burner

Reagents : Coal sample

Theory : The proximal analysis, which includes the determination of moisture, volatile matter, ash and fixed carbon this give quick and valuable information regarding for commercial and industrial use

Procedure : **Moisture** – It is determined by heating a known quantity of air dried coal from 105^{0}C-110^{0}C for one hour and calculating the loss in weight as percentage. Heat a silica crucible with lid, cool it in a desiccator and weigh. Take 1 gram of coal sample in it and again weigh, heat the crucible without lid in an air oven at 105^{0}C-110^{0}C for 1 hour, cool the crucible in a desiccator and weigh it again.

The loss of weight corresponds to the moisture.

Volatile Matter:- It is determined by heating 1 gram of air dried coal for 1 minute in a translucent silica crucible at a steady temperature of 925^{0}C in a muffle furnace.

Ash :- Take one gram of powdered air dried sample in previously weighed crucible having 5 cm diameter and 1 cm depth. Place a crucible on a clay pipe triangle and heat over a Bunsen burner with a wavy flame. Place the crucible without lid in muffle furnace at 750^{0}C and heat for 1 hour cool it in a desiccators to room temperature and weigh with lid.

Fixed carbon :- The sum of total of percentages of volatile matter, moisture and ash subtracted from 100 gives the percentage of fixed carbon

Calculation : **Moisture**

Weight of empty crucible = W1 gm

Weight of crucible + sample = W2 gm

Weight of crucible + sample after heating = W3 gm

$$\% \text{ moisture} = \frac{W_2 - W_3}{W_2 - W_1} \text{ X } 100$$

: **Volatile matter**

Weight of empty crucible = W_4 gm

Weight of crucible + sample = W_5 gm

Weight of crucible + sample after heating = W_6 gm

$$\% \text{ (moisture + volatile matter) } = \frac{W_5 - W_6}{W_5 - W_4} \text{ X } 100$$

$$\% \text{ volatile matter } = \left(\frac{W_5 - W_6}{W_5 - W_4} \text{ X } 100 \right) - (\% \text{ of moisture})$$

Ash

Weight of empty crucible = W_7 gm

Weight of crucible + sample = W_8 gm

Weight of crucible + ash = W_9 gm

$$\% \text{ Ash } = \frac{W_9 - W_8}{W_9 - W_7} \text{ X } 100$$

Fixed carbon

% Fc = 100 – (% Moisture + % Volatile matter + % Ash)

Precautions :

1. The apparatus should be thoroughly dried. There should be no trace of moisture inside the cup.
2. Work carefully.

Viva – voce :

1. Explain proximate analysis.
2. Explain ultimate analysis.

To determine the alkalinity of given sample of water using N/100 hydrochloric acid solution and methyl orange and phenolphthalein as indicators.

Experiment No. …………7…………. Date:…………………

Aim	:	**To determine the alkalinity of given sample of water using N/100 hydrochloric acid solution and methyl orange and phenolphthalein as indicators.**
Apparatus required	:	1. Burette 2.Pipette 3.Conical flask 4. Reagent bottles
Reagents	:	Phenolphthalein indicator, Methyl orange indicator, Hydrochloric acid solution (N/100),

Theory : Alkalinity of natural water may be attributed to the presence of salts of weak acids such as bicarbonates, phosphates, silicates and borates, which induces buffer capacity and resists the lowering of pH. Surface water-containing algae and also water treated by lime soda process may contain considerable quantities of carbonates and hydroxide alkalinity.

The alkalinity of water is due to the presence of hydroxide ion (OH$^-$), carbonate ion (CO3^{-2}) and bicarbonate ion (HCO3$^-$) present in the given sample of water. These can be estimated separately by titration against standard acid, using phenolphthalein and methyl orange as indicators. The chemical reaction involved can be shown by the equations given below

(A) $[OH-] + [H+] \rightarrow H2O$ P and M

(B) $[CO3^{-2}] + [H^+] \rightarrow HCO3^-$ ……………….. P and 1/2M

(C) $HCO3^- + [H^+] \rightarrow H2O + CO2$………………M

The titration of the water sample against a standard acid upto phenolphthalein end-point shows the completion of reactions [A] and [B] only. This amount of acid used thus corresponds to hydroxide plus one half of the normal carbonate present. The titration of the water sample against a standard acid to methyl orange end-point marks the completion of reaction [A], [B] and [C]. Hence the amount of acid used after the phenolphthalein end-point corresponds to one half of normal carbonate plus all the bicarbonates: while the total amount of acid used represents the total alkalinity (due to hydroxide, bicarbonate and carbonate ions)

The possible combinations of ions causing alkalinity in water are:

i. OH$^-$ only, CO3^{-2} only, HCO^{3-} only or

ii. OH$^-$ and CO3^{-2}, CO3^{-2} and HCO3$^{-\ t}$ogether

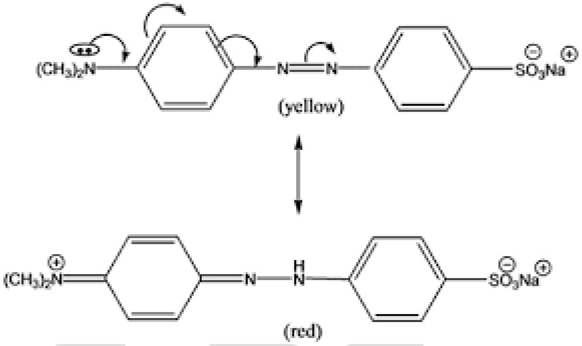
iii. The possibility of OH$^-$ and HCO3$^-$ ions together is not possible since they combine together to form CO3^{-2} ions

$$OH^- + HCO3^- \rightarrow CO3^{-2} + H2O$$

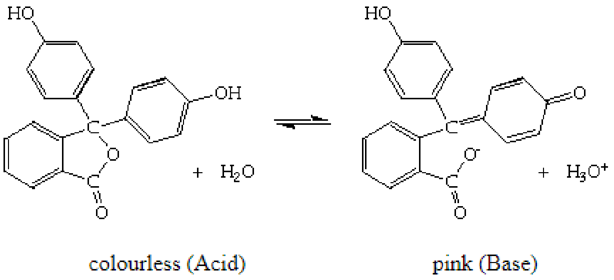
Procedure : **Alkalinity table**

Results of Titration to Phenolphthalein endpoint, (P) and Methyl orange end-point (M)	**Hydroxide**	**Carbonate**	**Bicarbonate**
[P] = 0	Nil	Nil	M
[P] = [M]	[P] or [M]	Nil	Nil
[P] = 1/2 [M]	Nil	2P	Nil
[P] > 1 /2[M]	2 (P) – (M)	2(M-P)	Nil
P < 1/2 [M]	Nil	2P	M-2P

Methyl orange is a valuable indicator for acid –base titration because it gives yellow color solution and red colour in acidic solution



Phenolphthalein is a triarylmethane dye but it is better known as an acid-base indicator



1. Rinse and fill the burette with N/100 HCl solution upto mark.

2. Pipette out 20 ml of the given water sample into 250 ml conical flask.

3. Then add 2-3 drops of phenolphthalein indicator to the sample (if solution turns colorless, the OH^- & $CO3^{--}$ alkalinity absent).

4. If the solution turns pink, titrate with N/100 HCl solution until colour change from pink to colorless.

5. Note the titrate value (Y), this is known as phenolphthalein end point.

6. Now add 2-3 drops of methyl orange indicator to the solution obtained in step 4.

7. If the colour turns pink, the $HCO3^-$ alkalinity is absent.

8. Otherwise continue the titration with N/100 HCl solution until the colour turns to reddish pink..

9. Record the titrate value (Z), this is known as methyl orange end point. Repeat the titration to get at least two concordant readings.

Observation Table : **Titration between given water sample and thiosulphate solution**

S. No.	**Volume of water sample taken (ml)**	**Burette reading (ml)**			**P end point (ml)**	**M end point (ml)**
		Initial (ml)	**With phenolphthalein (ml)**	**With methyl orange (ml)**		
1	20.0	0.0				
2	20.0	0.0				
3	20.0	0.0				

Calculation : **A.** If water sample contain OH^- and $CO3^-$

	OH^-	$CO3^{-2}$
[P] > 1 /2[M]	2 (P) – (M)	2(M-P)

Acid for neutralization of $CO3^{-2}$ alkalinity

2(M-P)
Normality of water with respect to $CO3^{-2}$ alkalinity

$N_1V_1 = N_2V_2$
(acid) = (water, $CO3^{-2}$)

N_1 = Normality of acid
V_1 = Vol. of acid for $CO3^{-2}$
N_2 = Normality of water = ?
V_2 = Vol. of water sample

$CO3^{-2}$ ion alkalinity in terms of $CaCO_3$ in g/l

Strength = N_2 x Eq. Weight of $CaCO_3$
= [N_2 x 50] g/l

$CO3^{-2}$ ion alkalinity in terms of $CaCO_3$ in ppm or mg/l

=[N_2 x 50 x 1000] mg/l

CO_3^{-2} ion alkalinity as CO_3^{-2} ion in ppm or mg/l

=[N_2 x 30 x 1000] mg/l

Normality of water with respect to OH^- alkalinity

$N_1V_1 = N_2V_2$
(acid) = (water, OH^-)

N_1 = Normality of acid
V_1 = Vol. of acid for OH^-
N_2 = Normality of water = ?
V_2 = Vol. of water sample

OH^- ion alkalinity in terms of $CaCO_3$ in g/l

Strength = N_2 x Eq. Weight of $CaCO_3$

= [N_2 x 50] g/l

OH^- ion alkalinity in terms of $CaCO_3$ in ppm or mg/l

=[N_2 x 50 x 1000] mg/l

OH^- ion alkalinity as OH^- ion in ppm or mg/l

=[N_2 x 17 x 1000] mg/l

Normality of water with respect to OH^- alkalinity

Results :

1. Methyl orange alkalinity:…………..

2. Phenolphthalein alkalinity:……………….

Precautions :

1 All the volumetric apparatus should be washed with distilled water before used.

2. Titration is carried out at room temperature.

3. Rinse the burette with a solution of Potassium dichromate solution taken in the burette.

4. Rinse the pipette with a solution to be taken in the pipette.

Viva – voce :

1. Write the cause of alkalinity..

2. Draw the structure of methyl orange in acidic medium

3. Draw the structure of methyl orange in basic medium

4. Draw the structure of phenolphthalein in acidic medium

To determine the amount of dissolved oxygen present in a given water sample .

Experiment No. ……8………. Date:…………………

Aim : **To determine the amount of dissolved oxygen present in a given water sample .**

Apparatus required :

1.Test tube
2. Test tube stand
3. Bunsen burner
4. 50ml Burette
5. 20 ml Pipette
6. 250 ml Conical Flask
7. 100 ml Measuring cylinder

Reagents : M_nSO_4 solution, Alkaline KI solution ,$Na_2S_2O_3$ solution, Concentrated H_2SO_4, Freshly prepared starch solution.

Theory : Water contains dissolved oxygen mainly due to the presence of aquatic living Organisms. The determination of dissolved oxygen in water is based on the Oxidation of KI to iodine . Then the liberated iodine is titrated against standard $Na_2S_2O_3$ solution. Here freshly prepared starch solution is used as indicaor.The reactions involved in it are as follows.

$$M_nSO_4 + 2KOH \blacktriangledown \quad M_n(OH)_2 + K_2SO_4$$

$$2M_n(OH)_2 + O_2 \blacktriangledown \quad 2M_nO(OH)_2$$

$$M_nO(OH)_2 + H_2SO_4 \blacktriangledown \quad M_nSO_4 + 2H_2O + [O]$$

$$2KI + H_2SO_4 + [O] \blacktriangledown \quad K_2SO_4 + H_2O + I_2$$

I_2 + starch ▼ Starch iodide (blue colourcomplex)

Procedure :

1.Take 500 ml water sample in a Stopper bottle.
2. Add 2 ml of M_nSO_4 and 5 ml of alkaline KI solution.
3. Shake the content and allow $M_nO(OH)_2$ (Brown Precipitate) to settle down.
4. Add 2 ml of concentrated H_2SO_4 and Shake.
5. Keep the Bottle for 10 min.
6.Take 100 ml of this and titrate slowly with standard $Na_2S_2O_3$ solution.
7.When colour of the solution becomes pale yellow add freshly prepared Starch solution
8. Again titrate with the standard solution $Na_2S_2O_3$ until colour changes from Blue to colourless .
9. Repeat to get three concordant readings.

Observation Table :

Titartion between sample solution and thiosulphate solution

S. No.	**Volume of sample taken (ml)**	**Burette reading**		**Volume of thiosulphate consumed**
		Initial	**Final**	
1				
2				
3				

Calculations : Applying normality formula, we have

N_1V_1 of sodium thisulphate = N_2V_2 of sample water

$$N_2 = \frac{N_1V_1}{V_2} = \frac{0.01 \times x}{100}$$

$$\text{Strength of oxygen} = 8 \times \frac{0.01 \times x}{100} \text{ g/L}$$

$$= 8 \times \frac{0.01 \times x}{100} \times 1000 \text{ mg/L}$$

Result : The amount of dissolve oxygen in water is found to be ....ppm

Precautions : 1. Experiment should be perform carefully.
2. Use chemical carefully

Viva - voice
1. What is the dissolve oxygen
2. What is end point?
3. Write the role of indicators.

To standarization of KM_nO_4 solution using standard oxalic acid solution

Experiment No. …09………… Date:…………………

Aim : **To standarization of KM_nO_4 solution using standard oxalic acid solution**

Apparatus required :

1.Test tube
2. Test tube stand
3. Bunsen burner
4. 50ml Burette
5. 20 ml Pipette
6. 250 ml Conical Flask
7. 100 ml Measuring cylinder

Reagents :

1. Oxalic acid solution(0.1N)
2. Sulphuric acid (10%)
3. KM_nO_4 solution

Theory : Potassium permanganate act as a stong oxidizing agent in acidic medium due to this property of KM_nO_4 can be titrate against standard reducing agent like disodium oxalate. Sulphuric acid is suitable for this purpose as it gives nascent oxygen.

$$2\ KM_nO_4 + 3\ H_2SO_4 \blacktriangledown\ K_2SO_4 + 2\ M_nSO_4 + 3\ H_2O + 5[O]$$

KM_nO_4 solution has pink colour. During the titration, M_n^{7+} of KM_nO_4 is reduced to colourless M_n^{2+} At the endpoint when the whole reducing agent is fully oxidised the additional excess drop of KM_nO_4 gives pink colour. Here KM_nO_4 acts as self indicator.

$$2M_nO_4 + 16\ H^+ + 5\ C_2O_4^{2-} \blacktriangledown\ 2\ M_n^{2+} + 8\ H_2O + 10\ CO_2$$

Procedure :

1.Rinse and fill the burette with the given KM_nO_4 solution.
2. pipette out 10 ml standard solution oxalic acid solution into a conical flask.
3. Add 10 ml of H_2SO_4 (10%) to it.
4.Carefully heat the solution flask to 60 – 70 ^{0}C if required some times not required.
5.Titrate it with the KM_nO_4 solution by drop wise addition till the colour changes from colourless to pink.
6.Take the initial and final burette reading. Note down the difference .
7.Repeatto get three concordant reading.

Observation Table :

S. No.	**Volume of sample taken (ml)**	**Burette reading**		**Volume of thiosulphate consumed**
		Initial	**Final**	
1				
2				
3				

Calculations :

Applying normality equation we have

$$N_1V_1 = N_2V_2$$

Where,

N_1 = normality of oxalic acid
V_1 = volume of oxalic acid
N_2 = normality of potassium permanganate
V_2 = volume of potassium permanganate

$$N_2 = \frac{N_1V_1}{V_2} = \frac{0.1 \times 10}{x}$$

$$\text{Strength of } KM_nO_4 \text{ solution} = \text{equivalent mass} \times \frac{0.1 \times 10}{X} \text{ g/L}$$

Result : the strength of KM_nO_4 solution is found to be ......g/L

Precautions :

1. Experiment should be perform carefully.
2. Use chemical carefully

Viva - voice

1. What is self-indicator
2. What is end point?
3. Write the role of indicators.

To determine the ferrous iron present in Mohrs salt by using standard KM_nO_4 solution

Experiment No. ……10………. Date:…………………

Aim : **To determine the ferrous iron present in Mohrs salt by using standard KM_nO_4 solution**

Apparatus required :

1.Test tube
2. Test tube stand
3. Bunsen burner
4. 50ml Burette
5. 20 ml Pipette
6. 250 ml Conical Flask
7. 100 ml Measuring cylinder

Reagents :

1. KM_nO_4 solution
2. Sulphuric acid (10%)
3. Mohrs salt solution

Theory : Mohrs salt is a double salt having formula $FeSO_4.(NH_4)_2SO_4.6H_2O$. Since potassium Permanganate acts as a strong oxidising agent in acidic medium so it can be used for the estimation of iron.When Mohrs salt is

titrated against KM_nO_4 solution, Fe^{2+} ions are converted to Fe^{3+} ions. Here,

$$5\,Fe^{2+} + M_nO_4^- + 8\,H^+ \blacktriangledown \qquad 5\,Fe^{3+} + M_n^{2+} + 4\,H_2O$$

Since KM_nO_4 decomposes quikly in presence of light, so its strength is not fixed for a long time. Hence, the strength of freshly prepared KM_nO solution is determined prior to the test.

Procedure :

1. Rinse and fill the burette with the given standard KM_nO_4 solution.
2. pipette out 10 ml Mohrs salt solution into a conical flask.
3. Add 10 ml of H_2SO_4 (10%) to it.
4. Titrate it with the standard KM_nO_4 solution by drop wise addition till the colour changes from colourless to pink
5. Take the initial and final burette reading. Note down the difference .
6. Repeatto get three concordant reading.

Observation Table :

S. No.	**Volume of sample taken (ml)**	**Burette reading**		**Volume of thiosulphate consumed**
		Initial	**Final**	
1				
2				
3				

Calculations :

Applying normality equation we have

$$N_1V_1 = N_2V_2$$

Where,

N_1 = normality of KM_nO_4
V_1 = volume of KM_nO_4
N_2 = normality of Mohrs salt
V_2 = volume of Mohrs salt

$$N_2 = \frac{N_1V_1}{V_2} = \frac{0.1\times x}{x}$$

$$\text{Strength of Mohrs salt solution} = \text{equivalent mass} \times \frac{0.1 \times 10}{10} \text{ g/L}$$

Result : the strength of KM_nO_4 solution is found to be ......g/L

Precautions :
1. Experiment should be perform carefully.
2. Use chemical carefully

Viva - voice
1. What is self-indicator
2. What is end point?
3. Write the role of indicators.