Government Girls' Polytechnic, Bilaspur

Name of the Lab: **Applied Chemistry Lab**

Practical : **Applied Chemistry Lab**

Class: **1st Semester (CSE, IT, ET&T)**

Teachers Assessment: 20 End Semester Examination: 100

EXPERIMENT – 1

AIM - Identification of two cations and two anions in a given sample of ore / powder/ mixture.

APPRATUS:-

Conical flask, test Tube, Reagent, spirit lamp, chemicals required.

OBSERVATION TABLE:-

(A) Table for Acid

Experiment	Observation	Inference
Salt + conc. H_2SO_4 + Heat	Smell of vinegar (CH_3COOH)	Conc. acid group present
Aqueous solution + Neutral $FeCL_3$	Brown ppt. or reddish colour evolved	CH_3COO^- present
Mix. + Conc. H_2SO_4 + Heat		Conc. acid group present
O.S. + MnO_2 + Conc.	Greenish yellow gas	Cl Present

H_2SO_4 + Heat		
O.S. + Na_2Co_3 extract + dil HNO_3 + Boil + $AgNo_3$	White ppt is formed	Cl Present
Chromyl chloride test- Mixture + K_2CR_2 + Conc. H_2SO_4 + Boil	Dark red vapour chromyl chloride as formed	Cl is confirmed.

(B) Table for Basic Radical

Experiment	Observation	Inference
Salt + NaoH Solution + Heat	Colourless gas, smell of NH_3	Zero group present
O. Solution + 1-2 drop of dil. HCL	White fumes evolved	NH_4^+ confirmed.
Original solution + dil. HCL + COOL	White precipitate is formed	1st group present
ppt. + H_2O + Boil Solution Divert solution into three parts:-		
1. 1st Part + KI Solution	Yellow ppt. (PbI_2)	Pb^{2+} may be present
2. 2nd Part + K_2CrO_4 Solution	Yellow ppt. ($PbCrO_4$)	Pb^{2+} may be present
3. 3rd Part + dil. H_2SO_4	White ppt. ($PbSO_4$)	Pb^{2+} may be present

RESULT:-

The given mixture are :-

Acid Radical	$CH_3\ COO^-$	CL
Basic Radical	NH_4	Pb^{2+}

PRECAUTION:

1. Take care by using conc. acid group.
2. Mixture should be taken less or sufficient quantity.

EXPERIMENT - 2

Object:-

To determine percentage of copper in a given sample by Brass titration.

Requirements:-

Test tube, test tube stand, Bunsen burner etc.

Observation Table:-

Table for Acid radicals

S. No.	Experiment	Observation	Inference
1.	Mixture + 1-2 ml of dil H_2So_4	Colourless, odourless gas with brisk effervescence, turns lime water milky.	Co_3^{2-} may be present.
2.	<u>Confirmation of Co_3^{2-}</u> salt solution + magnesium sulphate solution.	Formation of white precipitate in the cold.	Co_3^{2-} present.
3.	Mixture + conc. H_2So_4	No reaction	Conc. sulphuric acid group are absent.
4.	Boil a small amount of salt with dil HCl, filter the contents and to the filtrate add few drops of $Bacl_2$ solution.	A white ppt. is formed which is insoluble in conc. HCl.	SO_4^{2-} may be present.
5.	Leadacetate test: aqueous solution of salt + lead acetate solution	A white ppt. is formed, which is soluble in excess of hot ammonium acetate solution.	SO_4^{2-} confirmed.

Table for Basic radicals

S. No.	Experiment	Observation	Inference
1.	Mixture + NaOH + heat	No reaction	Zero group is absent.
2.	Original solution + dil HCl	Ppt. is not formed.	First group is absent.
	O.S. + dil HCl + heat + then pass H_2S gas from Kipp's apparatus.	Black ppt. is formed	Second group present.
4.	heat the black ppt. with 50% HNO_3 ppt. dissolves add dil H_2HSO_4 + alcohol	No white ppt.	Pb.$^{2+}$ is absent.
5.	To rest of the solution add NH_4OH in excess.	Blue coloured solution.	Cu^{2+} may be present.
6.	Confirmation- blue solution + CH_3COOH (acetic acid) + KI	A White ppt. is formed.	Cu^{2+} is confirmed.
7.	O.S. + conc. HNO_3 acid+boil + add NH_4Cl and boil again + cool the solution under tap water + add NH_4OH and shake.	reddish brown Ppt. is formed.	III group present (Fe^{3+} may be)

8.	Dissolve the reddish brown ppt. in dilute HCl, and divide the solution into two parts confirmation.		
	(I) Potassium ferocyanide test- to one part of the above solution add Pot. ferocyanide solution.	Prussian blue colouration.	Confirms Fe^{3+}
	(II) Potassium sulphocyanide test- to the second part add a little Pot. sulphocyanide solution.	Blood red colouration colouration.	Confirms Fe^{3+}

Result:

Acid radical : CO_3^{2-}, SO_4^{2-}

Basic radical : Cu^{2+}, Fe^{3+}

Precautions:

1) Take care by using acid group.
2) Mixture should be taken less or sufficient quantity. \

EXPERIMENT – 3

Aim:- To determine percentage of iron in given salt of iron (Ferrous ammonium sulphate)

Theory : Ferrous ammonium sulphate is a stable salt and can be oxidized easily with acidified $KMnO_4$ to the ferric salt as given by the equation.

$$2MnO4^{-} + 16H^{+} + 10Fe^{2+} \rightarrow 2Mn^{2+} + 10Fe^{3+} + 8H2O$$

as $2KMnO4 + 8H2SO4 + 10FeSO4 \rightarrow 2MnSO_4 + 5Fe_2 (SO_4)_3 + K_2SO_4 + 8H_2O$

Procedure:-

(A) Standardisation of $KMnO_4$ solution.

$KMnO_4$ solution is standardized by titrating it with standard Ferrous Ammonium Sulphate (FAS) solution. Standard solution of FAS (say N/30) in 100 ml is prepared by weighing out the requisite amount of the salt (1.307g) and dissolving in water acidified with dil H_2SO_4 to check hydrolysis. Now 10ml of this solution is taken in a conical flask and 5ml dil H_2SO_4 is added and titrated with $KnSO_4$ solution till a permanent light pink colour is obtained. The titration is repeated until three concordant readings are obtained from the normality a standard FAS solution, its volume and the volume of $KMnO_4$ used. The normality of $KMnO_4$ solution in calculated let it be N

(B) Determination of strength of unknown given Fe-salt (FAS) solution.

W g of the given FAS salt is dissolved in 100ml of water, acidified with dil H_2SO_4. 10 ml of the solution is now titrated with the same

$KMnO_4$ solution as above until permanent light pink colour in obtained. Titration is repeated to get concordant reading. Let the volume $KMnO_4$ solution used be V_1 ml.

$$= \frac{N_1 V_1}{V2} \times 392 = p \text{ g/l (say)}$$

i.e. 1000 ml FAS solution contains Pg of FAS

$$\therefore \text{ 100 ml FAS } = \frac{P \times 100}{1000} = \frac{P}{10} \text{ g}$$

Again 392 g FAS salt contains 55.85 g Fe

$$\therefore \frac{P}{10} \text{ g} \qquad \frac{55.85 \times P}{392 \times 10} \text{ g} = Q.g \text{ (say)}$$

i.e. Amount of Fe is the given FAS salt of Q.g.

Since W g of the given FAS salt contains Qg Fe

$$\therefore \text{ 100 g} \qquad \frac{Q \times 100}{w} \text{ g}$$

$$\text{\% of iron} = \frac{\text{wt of iron in sample}}{\text{wt of sample}} \times 100$$

i.e. percentage of Fe in the given FAS salt

$$= \frac{Q \times 100}{w}$$

$55.85 \times P \times 100$

$$= \frac{\qquad}{392 \times 10 \times w}$$

$$= \frac{55.85 \times N_1 \times V_1 \times 392 \times 100}{392 \times 10 \times w \times V2}$$

$$= \frac{N_1 \times V_1 \times 55.85 \times 10}{V2 \times w} \times 100$$

Observation Table

Standardization of $KMnO_4$ Soln. by N/30 FAS

S.No.	Reading of Burette		Vol. of FAS
	Initial	Final	
1.			
2.			

Concordant Vol. = …….. ml

Calculation (A) Standardization of $KMnO_4$ Solution

Volume of FAS solution taken = V

Normality of FAS solution = N

Volume of $KMnO_4$ used = V_1

So, Normality of $KMnO_4$ Solution , $N_1 = \frac{NV}{V_1}$

Observation Table

Standardization of Fe in the given unknown FAS salt

S.No.	Reading of Burette		Vol. of FAS
	Initial	Final	
1. 2.			

Concordant Vol. = …….. ml

(B) Determination of Fe in the given unknown FAS Salt :

Normality of $KMnO_4$ Solution = N_1
Volume of $KMnO_4$ used = V_1
Volume of FAS solution taken = V2
Normality of FAS = N2

Using, $N_1 V_1$ = N2V2 , the normality of FAS solution,

$$N2 = \frac{N_1 V_1}{V2}$$

Strength of FAS in g/l = Normality x Eq. wt.

EXPERIMENT – 4

Object:- To measure the pH of different solution.

Requirements:-

Digital pH meter, beaker, different solutions, 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.

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:-

1) 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 ^{O}C” 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 ^{O}C” control to the temperature of the selected buffer solution.

i) Set the function selector switch to pH position adjust 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:-

S. No.	Solutions	pH value
1.	Solution No. 1	4.5 pH
2.	Solution No. 2	5 pH
3.	Solution No. 3	6 pH
4.	un known	-----

Result:-

1) 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.

EXPERIMENT – 5

AIM - To determine the hardness of water in a given unknown water sample by EDTA method.

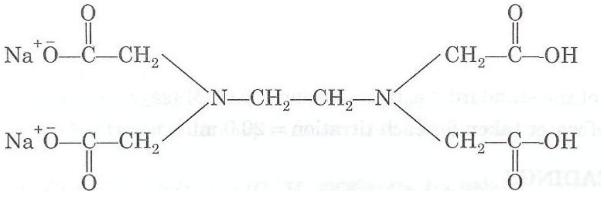
APPRATUS REQUIRED - Burette, pipette, conical flask, Beaker, test tube.

CHEMICAL REQUIRED -
1. EDTA solution
2. Water sample A & B
3. Erichrome Black – T (EBT)
4. Buffer solution of pH = 10

PROBLEM – Find out the hardness (in ppm) of given unknown sample 'B'. You are provided with a given intermediate solution of EDTA and standard water sample "A" which contains 1 gm of calcium carbonate dissolved in 1 litre of distilled water use erichrome Black-T as indicator.

THEORY -

The concentration of hardening ions in water can be determined by a titration technique, the titrant is the disodium salt of ethylene-diamine-tetraacetic acid



Disodium salt of ethylene diamine-tetracetic acid.

(Abbreviated as Na_2H_2Y or EDTA)

In aqueous solution, Na_2H_2Y dissociates into Na^+ and H_2Y^{2-} ions. Ca^{2+} and Mg^{2+} react with H_2Y^{2-} to form stable complexes in a solution having pH of about 10. A buffer solution containing ammonia and ammonium ions is used to maintain the pH of the solution around 10.

For the detection of the end point Erichrome Black T (EBT) is used as indicator. EBT forms complex ions with Ca^{2+} and Mg^{2+} ions, but binds more strongly to Mg^{2+} ions. Since only a small amount of EBT is added, only a small amount of Mg^{2+} ions is used in formation of complex and no Ca^{2+} ions are used. EBT indicator is sky-blue in solution but its complex with Mg^{2+} ions, $[Mg\text{-}EBT]^{2+}$, is wine-red.

$$\underset{}{Mg^{2+}(aq)} + \underset{\text{Sky-blue}}{EBT(aq)} \rightarrow \underset{\text{Wine-red}}{[Mg\text{-}EBT]^{2+}(aq)}$$

Thus, during titration when indicator is added to hard water, the initial colour is wine red. When the titrant is added, H_2Y^{2-} complexes with free Ca^{2+} and Mg^{2+} present in water and finally removes Mg^{2+} ions from the $[Mg\text{-}EBT]^{2+}$ complex ions. As a result the colour of the solution changes from wine-red to sky blue

$$\underset{\text{Wine-red}}{[Mg\text{-}EBT]^{2+}(aq)} + H_2Y^{2-}(aq) \rightarrow MgY^{2-}(aq) + 2H^{+}(aq) + \underset{\text{Sky blue}}{EBT(aq)}$$

It may be mentioned here that for the end point to appear, Mg^{2+} ions must be present in the solution. Therefore, a small amount of Mg^{2+} ions (as some salt) is added to the buffer solution and an equivalent amount of Na_2H_2Y is also added so that the added Mg^{2+} ions do not affect the amount of H_2Y^{2-} used during titration.

PROCEDURE -

1. Rinse Burette with EDTA solution & fill it upto the mark.

2. Rinse pipette with sample A solution & pipette out 10 ml of it into water washed conical flask.
3. Add about half tube of buffer solution having pH-10 to conical flask.
4. Add 2 or 3 drops of Erichrome Black T indicator of solution in conical flask. Wine colour is obtained.
5. Titrate solution with EDTA solution till the colour changes from wine red to original blue colour with single drops of EDTA solution.
6. Note down the reading.
7. Repeat the above process till the similar readings are obtained.

OBSERVATION :-

A. Sample “A” or known hard water

S.No.	Volume of Sample “A” (in ml)	EDTA of Burette reading		Volume of EDTA (in ml)
		Initial	Final	
1				
2				
3				

Calculation: - $N_1N_1 = N_2V_2$

B. Sample “B” or known hard water

S.No.	Volume of Sample “B” (in ml)	EDTA of Burette reading		Volume of EDTA (in ml)
		Initial	Final	
1				
2				
3				

Calculation: - $N_2N_2 = N_3V_3$

RESULT:- The total harness of given unknown water sample “B” is found to be ………. ppm

PRECAUTIONS:-

1. Reading should be taken carefully.
2. The end print should be checked.
3. Note that there should be no air bubble in nozzle of burette

EXPERIMENT – 6

AIM - Proximate analysis of a sample of Coal.

PRINCIPLE - 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 -

1. Moisture – It is determined by heating a known quantity of air dried coal from 105^0C-110^0C 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 105^0C-110^0C for 1 hour, cool the crucible in a desiccator and weigh it again.

   The loss of weight corresponds to the moisture.
2. 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 925^0C in a muffle furnace.
3. 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 claypipe triangle and heat over a Bunsen burner with a wavy flame. Place the crucible without lid in muffle furnace at 750^0C and heat for 1 hour cool it in a desiccators to room temperature and weigh with lid.
4. Fixed carbon :- The sum of total of percentages of volatile matter, moisture and ash subtracted from 100 gives the percentage of fixed carbon.

CALCULATION:-

1. Moisture –

Weight of empty crucible = W_1 gm

Weight of crucible + sample = W_2 gm

Weight of crucible + sample after heating = W_3 gm

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

2. 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

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

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

3. Ash –

Weight of empty crucible = W_7 gm

Weight of crucible + sample = W_8 gm

Weight of crucible + ash = W_9 gm

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

4. Fixed carbon :-

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

EXPERIMENT – 7

AIM - To determine the flash and fire point of a given oil by Penskey Martens flash point apparatus.

APPRATUS - Flash point apparatus.

THEORY - Flash Point :- "The lowest temp. at which the oil lubricants give enough vapours that ignite for a moment when a small flame is brought near it."

Fire point:- "The lowest temp. at which the vapours of the oil burn continuous for at last five second when a small flame is brought near it."

In most cases, the fire point are 5 – 40^{0}C higher than the flash point. A good lubricant has high flash and fire point and thus high working temperature.

DISCRIPTION OF THE APPRATUS-

Penskey Martens apparatus consists of a brass cup which is 5cm in diameter and 5.5 cm in depth. The level upto which oil fitted in cup is marked at about 3 cm below the top of the cup. The shutter provided at the top of the cup has a level mechanism when the shutter is turned. Opening for test flame and air are opened and the flame exposure device dips into the opening over the surface of the oil.

PROCEDURE:-

1. Thoroughly clean and dry all parts of the cup and its accessories before starting the test being sure to remove any solvent which had been used to clear the apparatus.

2. Support the tester on a level steady table.
3. Fill the cup with the sample to be tested to the level indicated by the filling mark.
4. Insert the thermometer, light the test flame and adjust to 4 mm in diameter.
5. The apparatus is heated, so that the oil temperature at use increase by about 5-6% per minute while the stirrer is rotated at approx 60 rotation/minute.
6. Record the flash point it is the temperature (µ) at the time when the test flame applied causes a distinct flash in the indicator of the cup.

RESULT:-

1. The flash point of given oil sample = $t_1{}^0C$
2. The fire point of given oil sample = $t_2{}^0C$

PRECAUTIONS:-

1. The oil cup should be dried.
2. The bulbs of thermometer should be inside the oil sample.
3. Breathing over the surface of the oil should be avoided.

EXPERIMENT – 8

AIM - To determine the calorific value of a solid fuel, using Bomb Calorimeter.

THEORY:-

The calorific value is the most important property of a fuel. The calorific value may be defined as the total quantity at heat, liberated by the complete combustion of a unit mass of the fuel. The calorific value determines the quantity of a fuel, and also helps in calculating thermal fuel, and a thermal efficiency and heat balance in the process where coal is used as a fuel.

HIGH OR GROSS CALORIFIC VALUE:-

The higher or GCV is defined as the total amount of heat liberated when one unit mass of the fuel has been completely burnt & the products of combustion have been cooled to room temperature. In such case water vapour produced by combustion of hydrogen and evaporisation of moisture coil get condensed and the heat is evolved. It is also taken into consideration. However, the heat evolved due to formation of H_2SO_4 and HNO_3 during combustion are subtracted from the heat evolved. The calorific value, as determined in the lab by bomb calorimeter represent the GCV.

LOWER OR NET CALORIFIC VALUE:-

In actual practice, the water vapour produced from hydrogen and moisture of the fuel during combustion is not condensed and escapes as such along with the hot combustic gases and hence a lesser amount of heat is available, hence, LCV or net calorific value may be defined as the net heat produced when unit mass of the fuel is burnt completely and products are allowed to escape.

LCV = GCV - [Latent heat of water vapour formed

DISCRIPTION :-

The calorific value of a solid or non-volatile liquid fuel is usually determined with the help of an oxygen bomb calorimeter.

The calorific value determination is carried out in a bomb calorimeter which consists of following parts as shown in figure

(i) The combustion bomb

(ii) The calorific vessel

(iii) The water jacket

(iv) Stirrer

(v) Thermometer

(vi) Crucible

(vii) Oxygen

(viii) Firing wire

WORKING:-

A known amount of sample (1gm) is burnt in a sealed chamber called bomb. The air replaced by pure O_2. Heat produced by burning the fuel must be equal to the amount of heat absorbed by the calorimeter before calculating the calorific value of a fuel with the help of bomb calorimeter. The water equivalent of the apparatus must be first determine.

CALCULATION:-

X = mass of fuel sample

W = Mass of water

w = Mass of water equivalent

t_1 = initial temperature of water

t_2 = Final temperature of water

L = HCV in cal./gm

Heat liberated by fuel – Heat absorbed by water

$$XL = (W+w)(t_2 - t_1)$$

$$\text{or } L = \frac{(W+w)(t_2 - t_1)}{X} \text{ cal/gm}$$

MATERIAL:-

Bomb calorimeter, given fuel sample, benzoic acid, burette, pipette, measuring, flask, N/10 NaOH, Analytical balance.

PROCEDURE:-

A known mass (about 0.5 to 1.0 gm) of given fuel is taken in clean & dry crucible. The crucible is then supported over the ring. A fine pt. wire, touching the fuel sample is then stretched across electrodes and cotton piece or thread of known wt. is tied to the pt. wire inserted in the sample inside. The core should be taken so that the lower end of the cotton thread touches the sample. Place 10 ml of distilled water inside the bomb with the help of pipette & place the cover in position and disconnect the O_2 supply. The bomb is then placed inside the vessel.

PRECAUTIONS:-

1. Do not use too much of the sample in any case. (Not more than 1 gm) Since bomb may not withstay for long-long time it effects for combustible change which liberate near than 10,000 calorie.
2. Do not change with more O_2 than necessary O_2 to obtain complete combustion and don't fire the bomb if an average of O_2 is advice.