



Roll Number: 47

Seat Number: \_\_\_\_\_

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### CERTIFICATE

Certified that Mr./Miss. Yash Savnang

Of D1AD FE semester **II** has successfully completed necessary experiments in **Engineering Chemistry-II** course under my supervision in VES Institute of Technology during academic Year **2020-2021**.

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**Vivekanand Education Society's Institute of Technology  
H & AS Department  
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Engineering Chemistry II [Lab 503]  
Sem II 2020 – 2021**

Outcomes: Learner will be able to...

1. Determine moisture and ash content of coal.
2. Analyze flue gas.
3. Determine saponification number and acid value of oil.
4. Determine flash point of a lubricating oil.
5. Synthesize a drug and a biofuel.
6. Determine Na/K & emf of Cu-Zn system.

Sr. N o.	Experiment Name	Date	Grad e	Page No.	CO Mappin g	Sign
1	Acid Value of Lubrication Oil	29/6		1	3	
2	Saponification Number of Oil	29/6		5	3	
3	Synthesis of Aspirin	29/6		9	5	
4	Demonstration of Electroplating	29/6		12	6	
5	Flame Photometry	29/6		14	6	
6	Determination of Moisture in Coal	29/6		19	1	

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## 1. ACID VALUE OF LUBRICATION OIL

AIM: To determine acid value of given oil sample.

APPARATUS: Pipette, burette, conical flask, round bottom flask, water condenser, volumetric flask, etc.

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## THEORY:

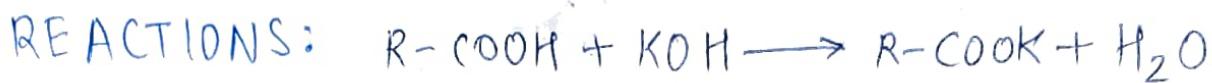
Acid value is defined as the number of milligrams of KOH required to neutralize free acids present in 1gm of oil. Freshly prepared organic oil contain negligible amount of free acid but on heating and on keeping the acid value or amount of free acids increases due to hydrolysis and oxidation of oil. The free acids present in oil impart sharp odour to oil and cause corrosion when present in a lubricating oil. In good lubricating oil, the acid should be less than 0.1. Increase in acid value should be taken as an indicator of oxidation of the oil which may lead to gum and sludge formation besides corrosion.

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## PROCEDURE:

Preparation of 0.1N Oxalic Acid:

Weigh accurately 0.63gm of Oxalic acid. Transfer the contents to 100ml beaker. Add water to dissolve Oxalic acid. Transfer the content to 1000ml volumetric flask and dilute up to the mark.



### OBSERVATION:

Standardisation of KOH:

Solution in burette	:	0.1 N KOH
Solution in conical flask	:	10 ml of Oxalic Acid
Indicator	:	Phenolphthalein
Pilot Reading	:	9-10 ml

Readings	I	II	III	constant
Initial (ml)	0.0ml	0.0ml	0.0ml	
Final (ml)	10ml	10ml	10ml	10ml
Difference	10ml	10ml	10ml	

Determination of acid value:

Solution in burette	:	0.1 N KOH
Solution in conical flask	:	25ml of oil
Indicator	:	Phenolphthalein
Pilot Reading	:	2 - 3 ml

Readings	I	II	III	Constant
Initial (ml)	0.0ml	0.0ml	0.0ml	
Final (ml)	2.5ml	2.5ml	2.5ml	2.5ml
Difference	2.5ml	2.5ml	2.5ml	

### Standardisation of KOH:

Fill the burette with 0.1N KOH solution. Pipette out 10ml of Oxalic acid solution to conical flask. Add 1 drops of phenolphthalein indicator. Titrate the solution with 0.1N KOH from the burette till solution turns pink.

### Determination of acid value:

weigh the given sample of oil accurately and place it in a round bottom flask. Add 50ml alcohol to it. Fix upon a water condenser to the round bottom flask and reflux it on a water bath for one and a half hour. Transfer the contents to a volumetric flask. Dilute the solution upto 250ml. Take 25ml of this diluted solution in a conical flask and titrate against 0.1N KOH using phenolphthalein as indicator.

RESULT: Acid value of the given lubricating oil  
is 2.8 mg of KOH.

## CALCULATION:

Standardisation of Oxalic acid:



∴ Normality of KOH  $[N_1] = 0.1$

Volume of KOH required for neutralization  
of oil = 2.5 ml

$$\text{Acid value} = \frac{\text{Volume of KOH required for neutralization}}{\text{Weight of oil sample}} \times \frac{\text{Normality of KOH} \times 56}{56}$$

$$= \frac{[2.5] \times [0.1] \times 56}{5}$$

$$= \underline{2.8 \text{ mg of KOH}}$$

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## 2. SAPONIFICATION NUMBER OF OIL

AIM: To determine the saponification number of oil sample.

APPARATUS: Round bottom flask, water condenser, conical flask, pipette, burette, etc

REAGENTS REQUIRED: Alcoholic KOH, 0.1N HCl, phenolphthalein indicator.

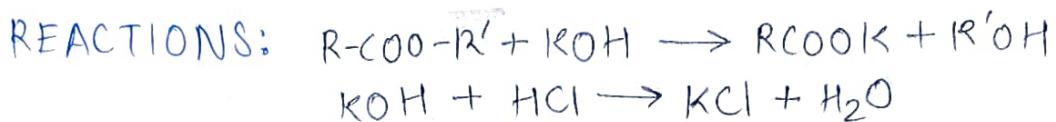
## THEORY:

Saponification number is defined as number of milligrams of KOH required to saponify 1g of organic oils of vegetable and animal origin which on hydrolysis with KOH gives glycerol and soap. The given oil sample is accurately weighed and placed in a round bottom flask. 25ml of KOH is added to it and the mixture is refluxed on a water-bath, after a water condenser is fixed to the round bottom flask. After refluxing for one and half hour, the heating is stopped and the contents of the flask are cooled and then transferred to a volumetric flask for diluting 250ml.

## PROCEDURE:

## PART - I:

Pipette out 25ml of KOH in a conical flask, add one drop of phenolphthalein indicator and titrate it against 0.1 N HCl from the burette till colour



### OBSERVATION:

#### PART - I

Solution in burette	:	0.1N HCl
Solution in pipette	:	25ml of KOH
Indicator	:	Phenolphthalein
End point	:	Pink to <del>red</del> colorless
Pilot reading	:	24-25ml

Readings	I	II	III	Constant x (cml)
Initial (cml)	0.0ml	0.0ml	0.0ml	
Final (cml)	25ml	25ml	25ml	25ml
Difference	25ml	25ml	25ml	

#### PART - II

Solution in burette	:	0.1N HCl
Solution in pipette	:	25ml Saponified oil
Indicator	:	Phenolphthalein
End point	:	Pink to colorless
Pilot reading	:	14-15ml

Readings	I	II	III	Constant y (cml)
Initial (cml)	0.0ml	0.0ml	0.0ml	
Final (cml)	15ml	15ml	15ml	15ml
Difference	15ml	15ml	15ml	

DATE:   |   |

changes from pink to colourless.

PART-II:

Pipette out 25ml of the diluted saponified oil in conical flask. Add a drop of phenolphthalein indicator to it and titrate the solution with 0.1N HCl from the burette till color changes from pink to colourless.

RESULT: Saponification number of oil sample is  
186.6 mg of KOH

## CALCULATION:

25ml of KOH → 25ml of 0.1N HCl (X ml)

25ml of saponified oil → 15ml of 0.1N HCl (Y ml)

Hence, Amount of KOH consumed for saponification →

$$(X - Y) \text{ ml of } 0.1\text{N HCl} = 10\text{ ml}$$

i.e. 10ml of 0.1N HCl → 10ml of 0.1N KOH

$$\text{Saponification number} = \frac{\text{Volume of KOH consumed} \times \text{Normality of KOH} \times 56}{\text{Weight of oil in grams}}$$

$$= \frac{10 \times 0.1 \times 56}{0.3}$$

$$= \underline{186.6 \text{ mg of KOH}}$$

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## 3. PREPARATION OF ASPIRIN

AIM: To prepare Aspirin from salicylic acid.

REQUIREMENTS: Salicylic acid, Acetic anhydride, Concentrated Sulphuric acid.

## THEORY:

Aspirin is the common name for the compound acetyl salicylic acid. It is one of the oldest and most common drugs in use today. It is both an analgesic (pain killer) and antipyretic (reduces fever). Its synthesis provides an insight that how chemistry is used in real-life application. To prepare aspirin, salicylic acid is reacted with an excess of acetic anhydride. A small amount of a strong acid is used as a catalyst which speeds up the reaction. In this experiment, phosphoric acid will be used as the catalyst. The excess acetic acid will be quenched with the addition of water. The aspirin product is not very soluble in water so the aspirin product will precipitate when water is added. Since acetic acid is very soluble in water, it is easily separated from aspirin product. The aspirin isolated in the step is crude product. A purified product can be obtained through recrystallization of crude product using ethanol. The purity of the product can be analyzed from its melting point (138-140°C).

REACTION: The synthesis reaction of aspirin is shown below:

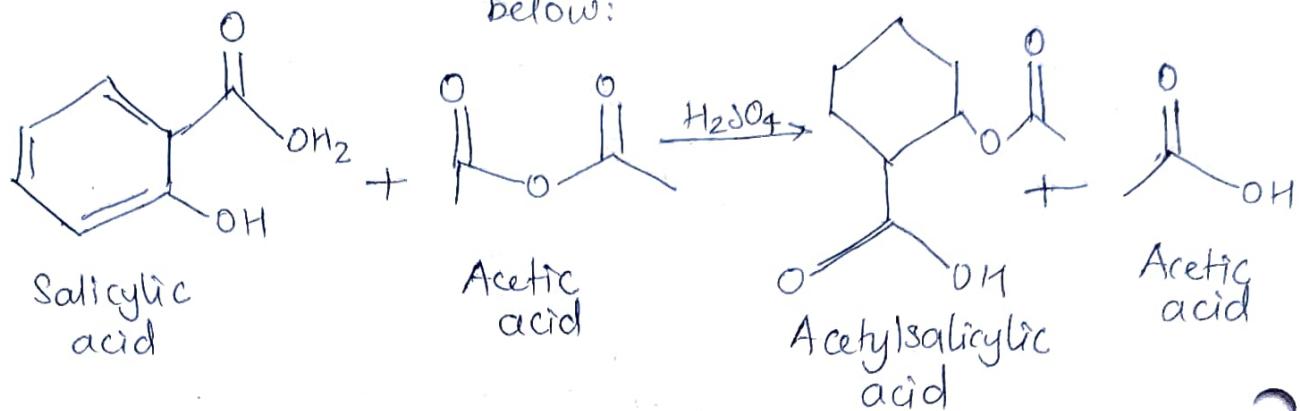


Fig: O-Acetylation of salicylic acid to give ~~sep~~ aspirin

#### OBSERVATIONS:

- weight of the product = 3.8gm

#### CALCULATIONS:

(i) Determination of theoretical yield:

weight of salicylic acid is 4gm

138gm of salicylic acid theoretically gives 180gm of aspirin

4gm of salicylic acid theoretically gives 5.2gm

(ii) Determination of practical yield:

Practical yield is 3.8gm

$$\text{(iii)} \quad \% \text{ Yield} = \frac{\text{Practical yield}}{\text{Theoretical yield}} \times 100 = \frac{3.8}{5.2} \times 100 = 70\%$$

### SAFETY CONSIDERATION:

The salicylic acid and aspirin may cause irritation to skin or eyes, but are basically not much hazardous. The acetic anhydride and sulphuric acid is corrosive can cause severe burns. Use them in hood in fan on mode. Ensure to wear gloves & safety goggles when using these chemicals.

### PROCEDURE:

- (i) In an absolutely dry 250ml conical flask, take provided (4g) salicylic acid.
- (ii) Add 5cm<sup>3</sup> of acetic acid anhydride to it in the hood, wear your goggles.
- (iii) Add 2 drops of conc. H<sub>2</sub>SO<sub>4</sub> stopper the flask & insert the thermometer.
- (iv) Keep the reaction mixture for 40 min in a water bath maintaining the temperature between 60-70°C
- (v) After 40 min cool the flask to room temperature. Place the flask in crushed ice-bath until the mixture has chilled and crystals have formed.
- (vi) Filter the solid aspirin through a piece of pre-weighed filter paper using a Buchner funnel, wash the crystal with deionized water & ethanol, dry & weight.

### RESULTS:

- (i) Theoretical yield : 5.2gm
- (ii) Practical yield : 3.8gm
- (iii) % yield : 70%
- (iv) Melting point of aspirin: 135°C

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#### 4. DEMONSTRATION OF ELECTROPLATING,

**AIM:** To apply a coating of Zn/Cu on iron strip by electroplating.

**APPARATUS:** Beaker, Battery eliminator, Iron strip, pure Zn/Cu strip,  $\text{ZnSO}_4/\text{CuSO}_4$  solution.

#### **THEORY:**

Electroplating is the process by which the coating metal is deposited on the base metal by passing electric current through an electrolyte solution, containing the soluble salt of the coating metal. The base metal is made cathode. Anode is the pure coating metal.

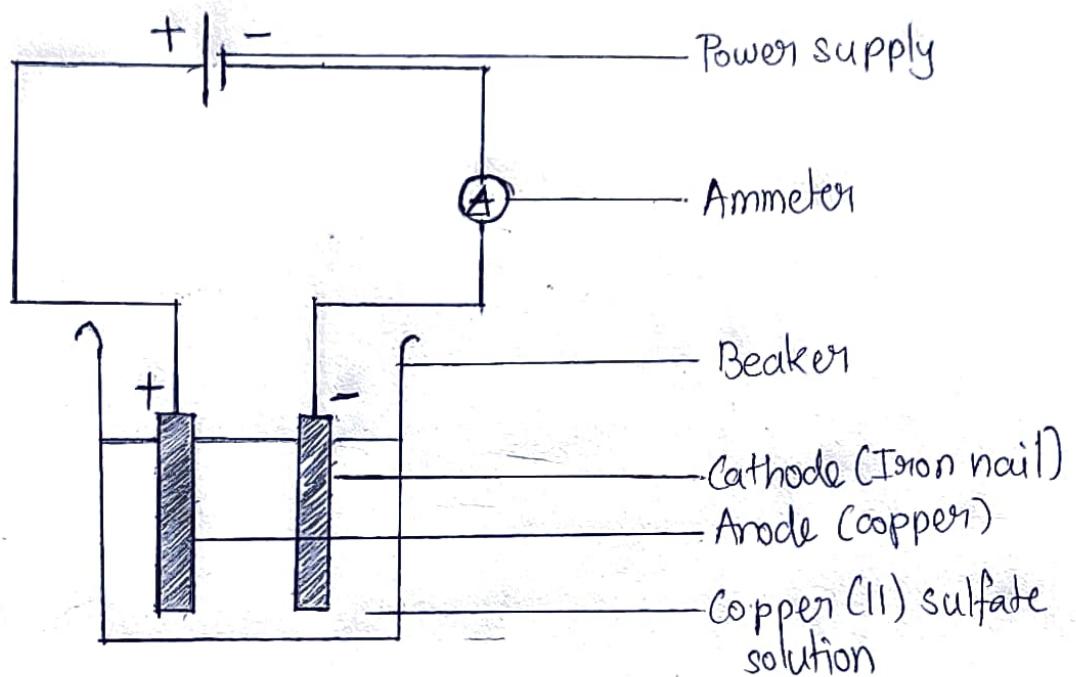
#### **METHOD:**

- (i) Prepare 1%  $\text{ZnSO}_4/\text{CuSO}_4$  solution in 500 ml beaker. Connect the iron strip (base metal) to the cathode.
- (ii) Connect the pure Zn/Cu strip to the anode.
- (iii) Adjust the battery eliminator to 2 volts.
- (iv) Place the two strips in the electrolyte solution in the beaker.
- (v) Observe the iron strip after 45 minutes.

**RESULT:** The uniform coating of Zn/Cu was deposited on the base metal that is iron strip.

OBSERVATION: A thin uniform coat of Zn/Cu was deposited on the iron strip.

CIRCUIT DIAGRAM:



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## 5. FLAME PHOTOMETRY

**AIM:** To determine potassium content of a commercial salt sample by flame photometry.

**REQUIREMENTS:** (i) A stock solution of K containing  $1 \times 10^3 \text{ g/cm}^3$  of the metal can be prepared by dissolving 0.477g of KCl in  $250 \text{ cm}^3$  of distilled water (strength=1000 ppm).  
(ii) Sample solution of K.

### **THEORY:**

Flame photometry is a traditional and simple method for determining Na/K concentration in biological fluids. It relies on the principle that an alkali metal salt drawn into a non-luminous flame will ionize, absorb energy from the flame and then eliminate emit light of a characteristic wavelength as the excited atoms decay to ground state. As the name suggests, flame photometric analysis counts the number of photons emitted per unit time at one particular wavelength from the flame in which a solution containing potassium ion is dispersed. The intensity of emission is proportional to the concentration of the element in the solution.

### **PROCEDURE:**

(i) Dilute  $10 \text{ cm}^3$  of the supplied stock solution of  $1 \times 10^3 \text{ g/cm}^3$  (1000 ppm) to  $100 \text{ cm}^3$  in a standard measuring flask with distilled water. This gives standard solution of 100 ppm.

## OBSERVATIONS:

Flask No.	Volume of standard solution in cm <sup>3</sup>	Final volume of solution in cm <sup>3</sup>	Concentration of solution in ppm	Emission intensity (I)
1	05	100	05	20
2	10	100	10	60
3	15	100	15	80
4	20	100	20	100
5	25	100	25	120
6	10 (sample)	100	Unknown	85

## CALCULATIONS:

Concentration of unknown from graph = 17.5 ppm

10 cm<sup>3</sup> of solution contains = 17.5 ppm

100 cm<sup>3</sup> of solution contains =  $10 \times 17.5 = 175$  ppm

175 ppm = 175 mg / 1000 cm<sup>3</sup>

Hence, 175 ppm = 17.5 / 100 cm<sup>3</sup>

Amount of K<sup>+</sup> in given sample is 17.5 mg

- (iii) Take six serially numbered  $100\text{cm}^3$  standard measuring flasks.
- (iv) Add  $5, 10, 15, 20$  and  $25\text{cm}^3$  of  $100\text{ppm K}$  solution to the standard measuring flask numbered from 1 to 5.  
Dilute each flask to  $100\text{cm}^3$  with distilled water.
- (v) Dilute the supplied sample solution to  $100\text{cm}^3$  in standard measuring flask with distilled water.
- (vi) Pipette out  $10\text{cm}^3$  of the diluted sample solution of K in  $100\text{cm}^3$  standard measuring flask and dilute up to the mark (flask no. 6).
- (vii) Measure the emission intensity for each solution and tabulate it.
- (viii) Plot the graph of concentration of solution (in ppm) against emission intensities. This forms the calibration curve.
- (ix) With the help of emission intensity of sample solution obtain concentration of K from the calibration curve.

#### RESULTS:

Amount of  $\text{K}^+$  in given sample =  $17.5\text{ mg}$

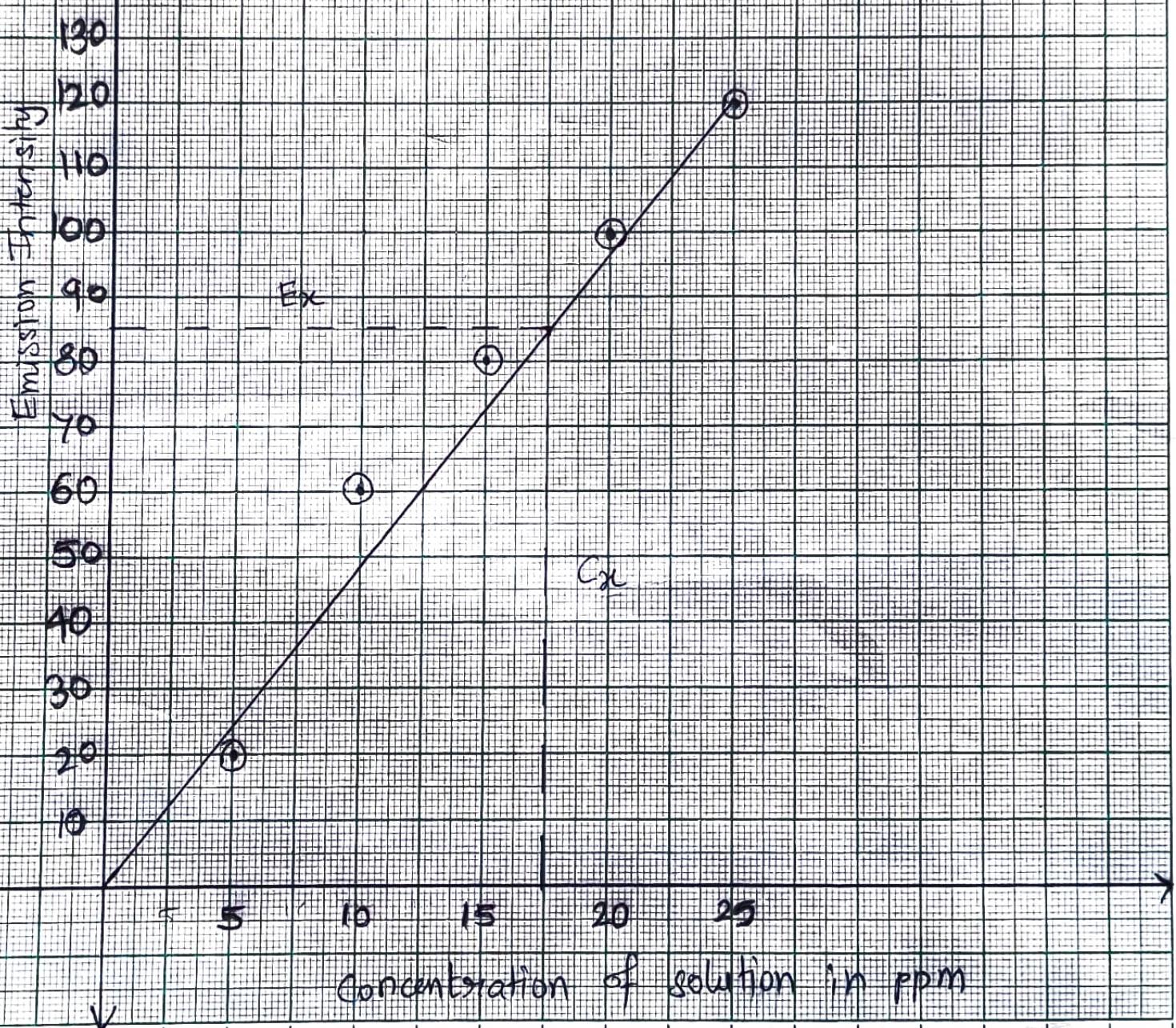
## GRAPH:

For the construction of calibration curve plot the graph of emission intensity for the solution against concentration of solution in ppm.

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Scale:

On X-axis: 1cm = 0.25 ppm  
On Y-axis: 1cm = 10 unit



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## 6. DETERMINATION OF MOISTURE IN COAL

**AIM:** To determine moisture in a given coal sample by proximate analysis.

**APPARATUS:** Silica crucible, Analytical weighing balance.

**REAGENTS:** Coal sample

### **THEORY:**

Coal is an important fossil fuel. It is formed by partial decay of plants buried under earth millions of years ago. The composition of coal varies widely depending upon its type, source and age. It is necessary to analyze the coal sample to determine its quality and thus the industrial use to which it can be put. The Proximate analysis is the simplest type of analysis of coal and it tells us about the partial utilization of coal. Moisture determination is a type of proximate analysis. The moisture which is lost on air drying is called surface moisture, it depends on the factors such as size of coal and its rank, treatment given to coal, nature of its surface. Moisture reduces its calorific value. Considerable amount of heat is wasted in evaporating the moisture during combustion. Further the cost is added up as water is brought and transported at the fuel price.

The analysis is carried out in accordance with standard specification (ASTM). The reproducible results can be obtained which can be utilized for assessing the application

## OBSERVATION AND CALCULATION:

Weight of empty crucible =  $X_1$  g = 21.25g

Weight of crucible + Sample =  $X_2$  g = 22.25g

Weight of crucible + Sample after heating =  $X_3$  g = 22.20g

$$\% \text{ Moisture} = \frac{(X_2 - X_3)}{(X_2 - X_1)} \times 100 = \frac{22.25 - 22.20}{22.25 - 21.25} \times 100 = 5$$

of coal for a particular industrial or domestic use.

#### PROCEDURE:

- (i) Weigh empty silica crucible on analytical balance accurately.
- (ii) Transfer approx 1-2 gms of coal sample to crucible and weigh.
- (iii) The crucible is kept inside the electric oven maintained at 105 to 110°C for an hour.
- (iv) After heating the crucible is removed with pair of tongs in desiccator for cooling.
- (v) On cooling is weighed on analytical balance.
- (vi) Difference in weight is reported in percentage as amount of inherent moisture in coal.

RESULT: Percentage Moisture present in coal sample = 5%.