

# LABORATORY MANUAL

**SEMESTER - FIRST**

**B.TECH**

**ENGINEERING CHEMISTRY**

Name of the Student \_\_\_\_\_

Registration No. \_\_\_\_\_ Batch No. \_\_\_\_\_ Branch \_\_\_\_\_



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(Approved by Director General of Civil Aviation, Govt. of India , All India Council for Technical Education, Ministry of HRD, Govt of India & Affiliated to Rajasthan Technical University. Kota, Rajasthan)



# School of Aeronautics (Neemrana)



## CERTIFICATE

This is to certify that Mr./ Ms. \_\_\_\_\_

Registration Number \_\_\_\_\_

of B.Tech ( ) has satisfactorily completed the term of the subject, Engineering Chemistry Lab, prescribed by Rajasthan Technical University, Kota.

Dated: \_\_\_\_\_

Signature of Faculty



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# EXPERIMENT-1

## PROXIMATE ANALYSIS OF SOLID FUEL

### OBJECT

To determine proximate analysis of solid fuel (coal) sample.

### APPARATUS REQUIRED

Electric oven, Muffle, Electrical or analysis or analytical balance, Desiccator, Silica crucible with lid and long legged tongs.

### REAGENTS REQUIRED

Given coal (solid fuel) sample.

### THEORY

Proximate analysis is the simplest type of analysis of coal and it provides us about the practical utilization of coal. It is the determination of moisture, volatile matter, ash content fixed carbon. This is called proximate analysis because the data collected vary with procedure adopted. This gives valuable information regarding commercial and probable fields of utility of the coal.

The analysis is an assay rather than true analysis, since the results have no absolute significance.

However, if the tests are carried out in accordance with standard specifications (British standard or American society for testing material (ASTM)). The results are reproducible which can be utilized for accessing the application of a fuel for probable use in particular industry.

### Moisture content

Higher percentage of moisture is undesirable because it increase the cost of coal as well as its transportation charges. Hence lesser the moisture content better the quality of coal as a fuel. Excess of moisture reduces the effective calorific value and quenches fire in furnace. However, presence of moisture, upto 10% produces a more uniform fuel bed and less of “fly-ash”.

Moisture content is the weight loss on heating and calculated in the percentage as

$$\% \text{ of moisture} = \frac{\text{Loss in weight}}{\text{weight of coal sample}} \times 100$$

**Volatile matter:** Volatile matter is not constituent of coal but it consists of a complex mixture of gas and liquid products resulting from the thermal decomposition of coal substance. If coal has high volatile content then a large proportion of fuel distill over as gas or vapor, a large proportion of which escapes unburnt.

So, higher volatile content in coal is undesirable. A high volatile matter containing coal burns with (i) a long flame (ii) high smoke and (ii) low calorific value.

Hence, lesser the volatile matter, better the rank of the coal.

Volatile matter is the weight loss on further heating and calculated as

$$\% \text{ of volatile matter} = \frac{\text{Loss in weight due to removal of volatile matter}}{\text{weight of coal sample}} \times 100$$

### Ash content

Ash is a non-combustible, useless matter which is left behind when all the combustible substance have been burnt off from coal. Ash usually consists of silica, alumina, iron oxide and small quantities of lime, magnesia etc. Its composition is of considerable in metal composition and consequently is a prime consideration in selecting the fuel.

It reduces the efficiency and calorific value of coal. It increases transporting, handling and storage costs. Lower the ash content, better the quality of coal.

$$\% \text{ of ash} = \frac{\text{weight of ash left}}{\text{weight of coal sample}} \times 100$$

### Fixed Carbon

The sum total of percentages of volatile matter, moisture and content, subtracted from 100 gives the percentage of fixed carbon. Higher the percentage of fixed carbon, greater the calorific value, smaller is percentage of volatile matter better the quality of coal. Hence, high percentage of fixed carbon is desirable.

Percentage of fixed carbon = 100 - Percentage of (Moisture + Volatile matter + content)

## PROCEDURE

### Determination of Content

1. Clean the silica crucible, dry and weight.
2. 1 gm of finely powdered coal sample taken in silica crucible and weight it again.
3. Keep the crucible with coal sample in the electric oven maintained at 110° C for an hour.
4. After heating, the crucible is taken out with the help of tongs, put into a desiccators for cooling.
5. Once it attains room temperature, again weight crucible.
6. The difference in weight is reported in percentage as the amount of internal moisture in coal.

### Determination of volatile matter

1. Take the dried sample after determining the moisture content and dried sample of coal covered with a lid a muffle furnace maintained at  $925 \pm 20^\circ \text{C}$  and heat about for 7-10 minutes.
2. After heating, the crucible is removed from muffle furnace with help of tongs.
3. Place the crucible on cooled iron plate to bring down its temperature and then transfer crucible in a desiccators so that it attains room temperature.
4. It is finally weight. Loss in weight corresponds to volatile matter content.

**Determination of Ash Content**

1. The residual coal in the crucible (from) is then heated inside a muffle furnace maintained at  $725 \pm 20^\circ\text{C}$  for half an hour.
2. The heating of crucible is done without covering it with lid.
3. After complete burning of coal remove the crucible with help of tongs.
4. The crucible is then cooled first in air and then inside desiccators.
5. The residue in crucible is reported as ash on percentage basis.

**Determination of fixed carbon**

The fixed carbon is calculated as percentage of fixed carbon =  $100 - (\text{Percentage of (Moisture + Volatile Matter + Ash content)})$

**Observations**

(i) Weight of the empty crucible =  $X_1$  g

(ii) Weight of crucible + sample =  $X_2$  g

(iii) Weight of crucible + sample, after heating at  $150^\circ\text{C}$  =  $X_3$  g

(iv) Weight of the crucible + sample after heating at  $925^\circ + 20^\circ\text{C}$  =  $X_4$  g

(v) Weight of the crucible + sample after heating at  $725^\circ + 20^\circ\text{C}$  =  $X_5$  g

**CALCULATION**

$$\% \text{Moisture} = \frac{(X_2 - X_3)}{(X_2 - X_4)}$$

$$\% (\text{Volatile matter} + \text{Moisture}) = \frac{X_2 - X_4}{X_2 - X_1} \times 100$$

$$\% \text{Ash} = \frac{(X_2 - X_5)}{(X_2 - X_1)}$$

**Fixed Carbon % age** =  $100 - (\text{Moisture} + \% \text{Volatile matter} + \% \text{content} + \% \text{Ash})$

**RESULT**

The given coal sample contains

- A. % Moisture = .....
- B. % Volatile matter = .....
- C. % Ash = .....
- D. % fixed carbon = .....



## QUESTIONS

Q1. Define a chemical fuel.

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Q2. What are fossil fuels and how they are formed?

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Q3. Give the name of three fossil fuels.

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Q4. What is meant by calorific value of a fuel?

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Q5. Define gross calorific value of a fuel.

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6. Why the Newton's are rings circular?

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Q7. What are the different varieties of coal formed inside the earth?

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Q8. Arrange the different varieties of coal in the decreasing order of their calorific value and their moisture contents.

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Q9. What are the steps of gravimetric analysis?

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Q10. What is meant by calorific intensity of coal?

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**Signature of Assistant Professor**

**Signature of C.I. / Principal**

# **EXPERIMENT- 2**

## **CALORIFIC VALUE OF SOLID FUEL**

### **OBJECT**

To determine the calorific value of solid fuel using Bomb calorimeter.

### **APPARATUS REQUIRED**

Bomb calorimeter.

### **REAGENTS REQUIRED**

Solid/liquid fuel under test.

### **THEORY**

A known mass or volume of the fuel is burnt completely and the amount of heat generated is absorbed by water and measured. Then the quantity of heat produced by burning a unit mass of unit volume of the fuel is calculated. The calorific value of the fuel can be expressed in two ways:

#### **(a) Gross or higher calorific value (HCV)**

Hydrogen is found to be present in almost all fuels and when the calorific value of hydrogen-containing fuel is determined experimentally, the hydrogen is converted into steam. If the products of combustion are condensed to the room temperature, the latent heat of condensation of steam also gets included in the measured heat, which is then called higher or gross calorific value.

#### **Definition**

It is defined as the total amount of heat generated when a unit mass or unit volume of fuel is completely burnt and the products of the combustion are cooled down to room temperature (i.e. 15°C or 60°F).

#### **(ii) Net or lower calorific value (LCV)**

In actual use of any fuel, the water vapor and moisture etc. are not condensed and escape as such along with hot combustion gases. Hence, a Lesser amount of heat is available.

#### **Definition**

It is defined as the heat Produced when unit volume of the fuel is completely burnt and the products of combustion are allowed to escape. Thus Net calorific value =Gross calorific value.

(LCV) =(HCV) - Latent heat of water vapor produced

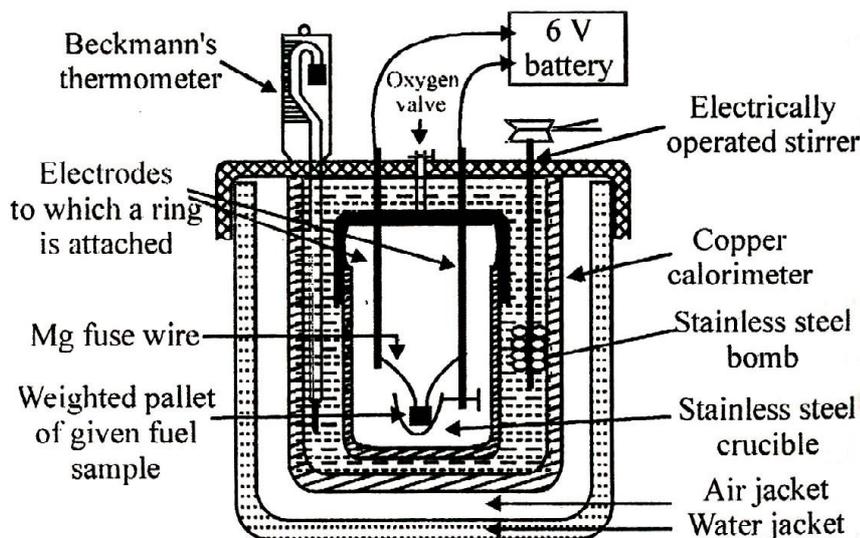
LCV = Gross C.V. - Mass of hydrogen X 9 X latent heat of steam

Because 1 part by mass of hydrogen produces 9 parts (1+8) by mass of water and latent heat of steam is 587 kcal.

Bomb calorimeter is an apparatus used to determine the calorific value of solid and liquid fuels.

## CONSTRUCTION

Bomb calorimeter consists of a stainless steel vessel, called bomb which is provided with a screw type lid, so as to make a perfect air-tight seal. The lid, in turn, is provided with two electrodes which can be connected to a 6 V battery. One of the two stainless steel electrodes is attached with a small ring which acts as a support for the crucible. The bomb is placed in a copper calorimeter having a known weight of water. The copper calorimeter is surrounded by an air jacket and a water jacket to prevent heat loss due to radiation. The calorimeter is also provided with an electrical stirrer and a Beckman's thermometer which can read accurately at temperature difference up to  $1/100^{\text{th}}$  of a degree.



## PROCEDURE

1. Take accurately weighed amount of fuel is placed in the silica crucible. The crucible is then supported over the ring.
2. A fine Mg wire touching the fuel sample is then stretched across the electrodes.
3. Bomb lid is tightly screwed and then the oxygen supply is forced into the bomb with the help of oxygen inlet valve till the pressure is reached 25-30 atm.
4. Place the bomb inside the copper calorimeter containing a known mass of water start the stirrer and the initial temperature of the water is noted after thorough stirring.
5. The electrodes are connected to 6V Battery and circuit is then completed. The sample burns and heat is liberated which is transferred to water. Uniform stirring of water is continued throughout the experiment by the electric stirrer.
6. The maximum temperature shown by Beckman's thermometer is recorded. The calorific value of the fuel can now be calculated.

**OBSERVATIONS**

- (i) Weight of the fuel taken in the crucible = x gm
- (ii) Weight of the water taken in calorimeter = W gm
- (iii) Water equivalent of calorimeter, stirrer, thermometer, bomb etc. = w gm (water equivalent of apparatus = weight of apparatus x specific heat)
- (iv) Initial temperature of water in calorimeter =  $t_1$  °C
- (v) Final temperature of water in calorimeter =  $t_2$  °C
- (vi) The higher calorific value of the fuel = L cal/gm

**CALCULATIONS**

Heat liberated by burning of fuel = xL cal

Heat absorbed by water =  $[W \times S \times (t_2 - t_1)]$

Heat absorbed by apparatus =  $[W \times S \times (t_2 - t_1)] = [w \times (t_2 - t_1)]$

Thus total heat absorbed by water, apparatus etc.

$$= [W + w] \times (t_2 - t_1)$$

But heat liberated by the fuel = heat absorbed by water, apparatus etc.

$$= xL = (W + w) \times (t_2 - t_1)$$

$$HCV \text{ of fuel (L)} = \frac{(W + w)t_2 - t_1}{x} \text{ cal / gm}$$

The water equivalent of calorimeter (w) is determined by burning a fuel of known calorific value.

The fuel used for this purpose are benzoic acid (HCV= 6325 kcal/kg) and naphthalene (HCV = 9,688 kcal/kg).

If H= % of hydrogen in fuel then,  $9H/100$  gm = mass of water from one gm of fuel = 0.09H gm  
So, heat taken by water forming steam = 0.09H x latent heat of steam (587 cal/gm)

LCV=HCV - latent heat of steam (587 cal/gm) =HCV - latent heat of vaporization of water formed

$$LCV = (HCV - 0.09 H \times 587) \text{ cal/gm}$$

**Correction**

However, to get more accurate results, the following corrections must be considered in the above equations.

**(i) Fuse wire correction**

The heat produced, as measured above, includes the heat given out by ignition of the fuse wire used. Hence it must be subtracted from the total value.

**(ii) Acid correction**

The fuel containing N and S are oxidized, under high pressure and temperature of the ignition, to nitric acid and sulphuric acid respectively.

Formation of these acids are exothermic reactions. So, the measured heat also includes the heat given out during the acid formation.

**(iii) Cooling correction**

If the time taken to cool the water in calorimeter from maximum temperature to room temperature is noted i.e. the rate of cooling (dt/minute) and the actual time taken for cooling (t minutes), the cooling correction of dt x (t) is added to the rise in temperature

$$(HCV) = [(W + w) \times (t_2 - t_1 + \text{Cooling correction})] - \frac{[\text{Acid} + \text{Fuse wire corrections}]}{\text{Mass of fuel (x)}}$$

**RESULT**

(i) Higher calorific value of given sample is.....cal/gm.

(ii) Lower calorific value of given sample is.....cal/gm.

**PRECAUTIONS**

1. Solid sample should be taken in form of pallet for combustion.
2. Oxygen pressure should not be high.
3. Only one gram of sample should be taken for more accurate results.
4. Pallet must be completely burnt during combustion.

**CONCLUSION**


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**QUESTIONS**

Q1. Define calorific value. For what purpose bomb calorimeter is used?

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Q2. What are the units of calorific value?

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Q3. Name different varieties of coal.

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Q4. Define higher calorific value or gross calorific value?

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Q5. Write down the formula for low calorific value?

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Q6. Why is the water taken into the bomb calorimeter?

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Q7. What are the various corrections made in the determination of calorific value of bomb calorimeter?

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Q8. How will you define the lower calorific value?

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Q9. Why we subtract the fuse wire correction and acidic correction in the calculation of calorific value by bomb calorimeter?

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Q10. Give the formula for High calorific value.

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# EXPERIMENT- 3

## STRENGTH OF FERROUS AMMONIUM SULPHATE

### OBJECT

To determine the strength of ferrous ammonium sulphate (FAS) solution with the help of  $K_2Cr_2O_7$  solution.

### APPARATUS REQUIRED

Conical flask, Burette, Pipette, Glass rod, White glazed tile, Measuring cylinder etc.

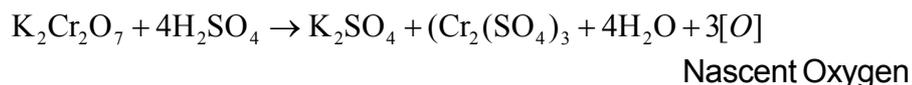
### REAGENTS REQUIRED

Potassium dichromate solution (N/20), FAS (unknown), Dil.  $H_2SO_4$ , Potassium ferricyanide (indicator) etc.

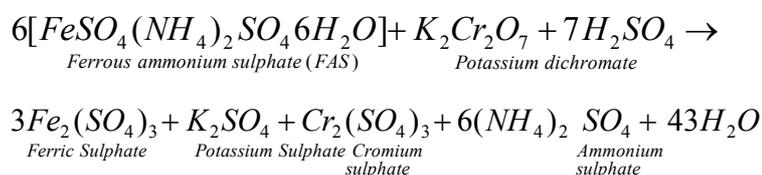
### THEORY

Chemical equation can be expressed as follows:

$K_2Cr_2O_7$  is a good oxidizing agent, and in the presence of dil.  $H_2SO_4$  it liberates three atoms of oxygen.



This liberated nascent oxygen oxidize  $Fe^{+2}$  ions of FAS into  $Fe^{+3}$  ions, as expressed :



When reaction is completed or all the  $Fe^{+2}$  ions get oxidize in to  $Fe^{+3}$  ions, then they will not form any complex with indicator as it also contain  $Fe^{+3}$  ions. Therefore, at the end, there is no blue-green coloration.

### PROCEDURE

1. Rinse the glass wares with water properly.
2. Fill the burette with potassium dichromate Solution (N/20) and note the initial reading carefully by reading upper meniscus of burette.
3. Pipette out 10 ml of unknown FAS solution into conical flask, add 10 ml of dil.  $H_2SO_4$  by measuring cylinder.
4. With the help of glass rod, arrange few drops of indicator on the white glazed tile (As shown in figure).
5. Titrate the solution with  $K_2Cr_2O_7$  dropwise with continuous shaking.

6. After addition of successive 1 ml of titrant. test the titrating mixture with a drop of indicator on white glazed tile with the help of glass rod. It will show blue-green color.
7. When the reaction is completed, indicator will not show blue-green color.
8. Note the burette reading at this point.
9. For example, upto addition of 9ml of dichromate solution, indicator gives blue-green color but at the addition of 10ml, it does not give color. It means, the end point lies between 9-10 ml. Then in next titration, check the exact end point between 9-10 ml of burette reading by dropwise addition.
10. Repeat the procedure to get concordant readings.  
As an example, it may change according to concentration.  
1-9 : Successive addition of 1 ml of  $K_2Cr_2O_7$  solution.

### OBSERVATION TABLE

S. No.	Volume of FAS solution in conical flask (ml)	(Burette reading) Volume of $K_2Cr_2O_7$ (ml)		Difference in burette reading (ml)	Concordant reading (ml)
		I.B.R	F.B.R		
1	10.0				
2	10.0				
3	10.0				

### CALCULATIONS

By using normality equation

$$N_1 V_1 = N_2 V_2$$

$$(FAS) \quad (K_2Cr_2O_7)$$

$N_1$  = Normality of FAS (unknown)

$V_1$  = Volume of FAS (10 ml)

$N_2$  = Normality of  $K_2Cr_2O_7$  (N/20)

$V_2$  = Volume of  $K_2Cr_2O_7$  (concordant reading)

$$\therefore N_1 = \frac{N_2 V_2}{V_1}$$



**QUESTIONS**

Q1. How many methods are available in redox titration for determining the strength of ferrous ammonium sulphate?

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Q2. What do you mean by internal indicator method?

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Q3. Define external indicator?

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Q4. Give one example of external and internal indicator.

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Q5. Explain oxidizing nature of potassium dichromate in presence of sulphuric acid with the help of chemical equations.

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Q6. Why the green color appears when  $K_2Cr_2O_7$  is titrated with FAS in acidic medium?

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Q7. Why dilute sulphuric acid is added while preparing standard solution of Mohr salt.

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Q8. Why ferrous sulphate isn't used instead of ferrous ammonium sulphate?

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Q9. Why dilute  $H_2SO_4$  and not  $HCl$  &  $HNO_3$  is used in redox titration?

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Q10. What are the indicators used in  $K_2Cr_2O_7$  titration?

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# EXPERIMENT - 4

## STRENGTH OF $\text{CuSO}_4$ SOLUTION

### OBJECT

To determine the strength of  $\text{CuSO}_4$  solution with the help of hypo solution.

### APPARATUS REQUIRED

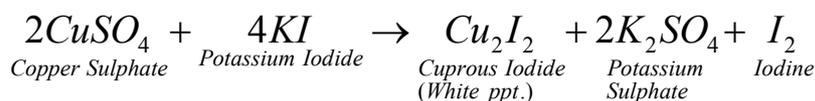
Burette, Pipette, Iodometric flask, Measuring cylinder.

### REAGENTS REQUIRED

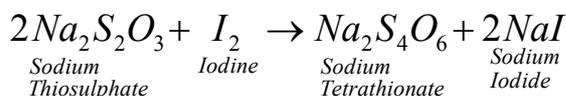
Sodium thiosulphate (N/20) solution, Potassium iodide (10%) solution, Freshly prepared starch.

### THEORY

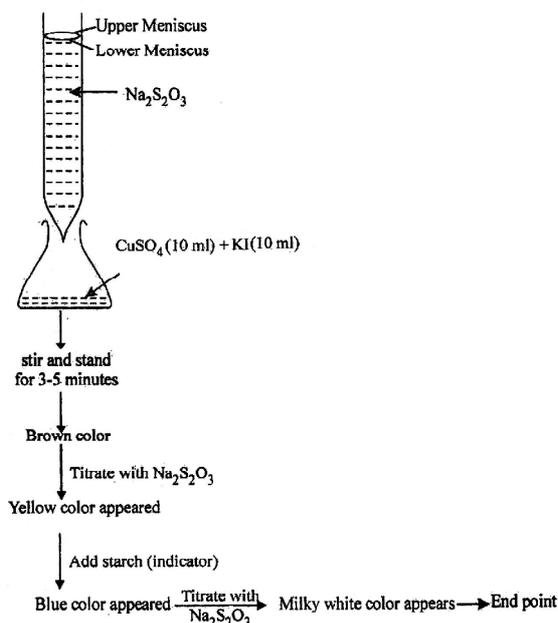
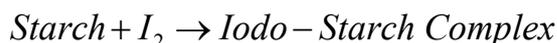
For determination of copper sulphate ( $\text{CuSO}_4$ ), solution of potassium iodide (KI) is added of  $\text{CuSO}_4$  solution. It gives white precipitate of cuprous iodide ( $\text{Cu}_2\text{I}_2$ ), potassium sulphate ( $\text{K}_2\text{SO}_4$ ) and equivalent amount of iodine ( $\text{I}_2$ ) is liberated.



This free iodine is titrated against standard solution of sodium thiosulphate or hypo ( $\text{Na}_2\text{S}_2\text{O}_3$ ), using starch as indicator. This iodine is reduced to sodium iodide (NaI) and hypo converts into sodium tetrathionate.



Iodine also with starch and form blue colored iodo-starch complex.



Due to this, starch is added to reaction mixture just before the completion of reaction between

sodium thiosulphate and iodine should be available to starch for indicating completion of reaction. As the reaction is completed or iodine is reduced to sodium iodide, the blue color of iodo-starch complex get disappeared and show the white color of cuprous iodide. It indicates about the end point.

Hence, the relation between chemicals are:



For this titration, neutral medium is compulsory therefore, presence of any mineral acid is avoided. If it is present then it should be neutralized by adding sodium carbonate solution.

## PROCEDURE

1. Rinse the glass wares properly with distilled water.
2. Fill the burette with N/20 sodium thiosulfate solution.
3. Pipette out 10 solution in iodometric flask.
4. Stoppered the iodometric flask and keep it for 3-5 minutes in dark. After that, the solution turns to brown.
6. Titrate this solution in iodometric flask until the brown color fades to yellow.
7. At this points of appearance of yellow color, add 1 ml of starch as indicator. The solution turns to blue.
8. Continue titration with hypo till the disappearance of blue color with the appearance of milky white color. It indicates the end point.
9. Note the burette procedure to get concordant readings.

S. No.	Volume of $\text{CuSO}_4$ taken in iodometric flask (ml)	Burette reading Volume of hypo consumed I.B.R F.B.R	Difference in burette reading (ml)	Concordant reading (ml)
1	10.0			
2	10.0			
3	10.0			

## CALCULATIONS

Using normality equation:

$$\begin{aligned}
 N_1 V_1 &= N_2 V_2 \\
 (\text{CuSO}_4) & \quad (\text{Hypo}) \\
 N_1 &= \text{Normality of CuSO}_4 \text{ (unknown)} \\
 V_1 &= \text{Volume of CuSO}_4 \text{ (10ml)} \\
 N_2 &= \text{Normality of hypo (N/20)} \\
 V_2 &= \text{Volume of hypo (burette reading)}
 \end{aligned}$$



**QUESTIONS**

Q1. Why titration mixture ( $\text{CuSO}_4$  & KI) should be kept for five minutes in iodometric flask before titration?

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Q2. Write the complete chemical equation when  $\text{CuSO}_4$  is reacted with hypo in presence of KI.

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Q3. Name various types of titrations known to you.

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Q4. What type of titration is of  $\text{CuSO}_4$  vs  $\text{Na}_2\text{S}_2\text{O}_3$ ?

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Q5. What is the difference between iodimetry and iodometry?

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Q6. Why the freshly prepared solution of starch is formed?

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Q7. Why is the distilled water always used in preparation of solutions?

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Q8. Why the hot solution not poured in the burette?

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Q9. Why a burette with rubber tap should not be used in iodimetry?

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Q10. Why excess of KI is used always?

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## EXPERIMENT - 5

### STRENGTH OF SODIUM HYDROXIDE AND SODIUM CARBONATE

#### OBJECT

To determine the strength of sodium hydroxide and sodium carbonate in an alkali mixture.

#### APPARATUS REQUIRED

Burette, Pipette, Conical flask, Measuring flask.

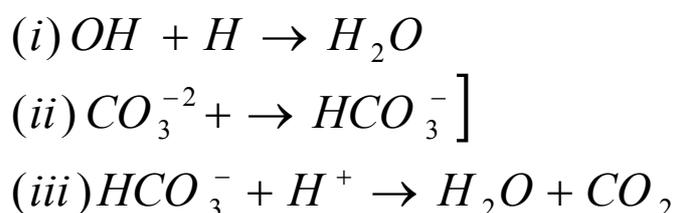
#### REAGENTS REQUIRED

Phenolphthalein, Methyl orange, HCl (N/10), alkali water sample, standard  $\text{Na}_2\text{CO}_3$  solution (N/20).

#### THEORY

As it already discussed that alkalinity of water is caused by hydroxide ions ( $\text{OH}^-$ ), carbonate ions ( $\text{CO}_3^{2-}$ ) and bicarbonate ions ( $\text{HCO}_3^-$ ). These can be estimated separately by titrating against standard acid using phenolphthalein and methyl orange as internal indicator.

Involved chemical reactions are given below



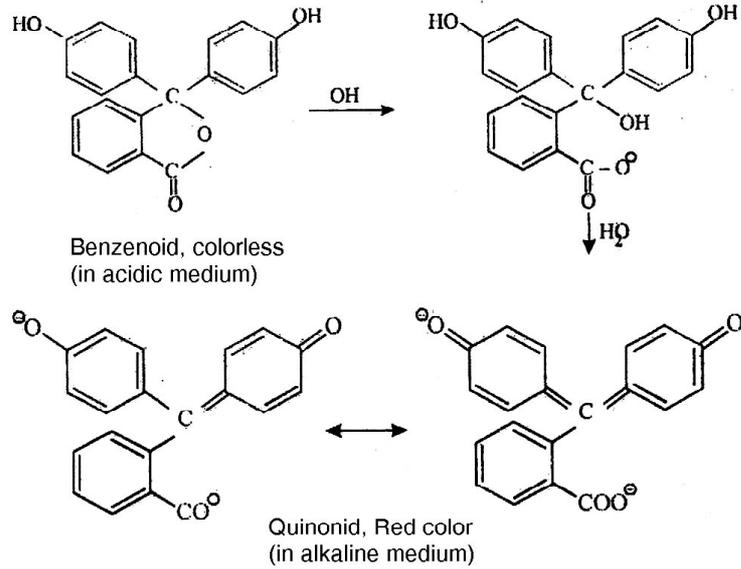
Phenolphthalein indicates about the completion of reactions (i) and only whereas methyl orange indicates the completion of all three reactions i.e. (i), (ii) and (iii). Hence, the amount of acid used after phenolphthalein end point corresponds to one half of the carbonate ions and all the hydroxide ions. Total amount of acid indicates the total alkalinity.

Reaction (i) and completed in the pH range of 8-10 therefore phenolphthalein is used for this purpose. Reaction (iii) completed in the pH range of 3-4 which can be easily determined using methyl orange indicator.

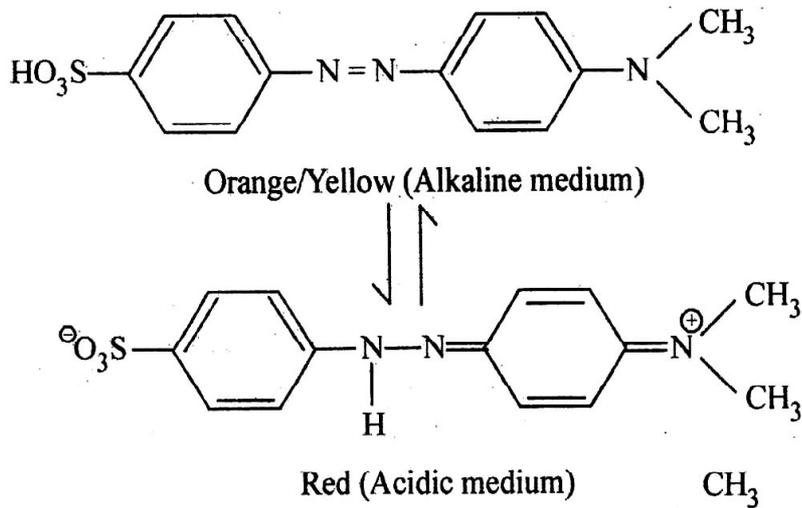
These three alkalinity causing ions may be present in water in various combinations like:

- (i) Only  $\text{OH}^-$ , only  $\text{CO}_3^{2-}$  and only  $\text{HCO}_3^-$  ions.
- (ii)  $\text{OH}^-$  and  $\text{CO}_3^{2-}$  and only  $\text{HCO}_3^-$  together.
- (iii)  $\text{OH}^-$  and  $\text{CO}_3^{2-}$  ions cannot remain together as they form  $\text{H}_2\text{O}$  and only  $\text{CO}_3^{2-}$  ions  
$$\text{OH}^- + \text{HCO}_3^- \rightarrow \text{H}_2\text{O} + \text{CO}_3^{2-}$$

Phenolphthalein and methyl orange both act as acid-base internal indicator. They show different resonating structures in different medium having varying colors.

**Example:** Forms of Phenolphthalein

Similar methyl orange has following resonating structures:



Therefore, in this titration at phenolphthalein and point, red color changes in to colorless and at methyl orange and point, the color changes yellow to red.

## PROCEDURE

### (A) Standardization of HCl

1. Wash the glass wares properly.
2. Fill the burette with HCl and not the initial reading by lower meniscus as HCl is colorless solution.
3. Take 10 ml of standard sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) solution in conical flask and add 2-3 drops of methyl orange as an indicator.
4. Titrate the solution with HCl till the color changes from yellow to red. it indicates end point note the burette reading carefully.
5. Repeat the experiment for getting concordant readings.

### (B) Determination of $\text{Na}_2\text{CO}_3$ and NaOH in a sample.

1. Burette is already filled with HCl
2. Take 25 ml of sample into conical flask add 2-3 drops of phenolphthalein. The solution turns to pink.
3. Titrate it with HCl drop wise and with continuous stirring till the color changes from pink to colorless. it indicates phenolphthalein end point (P). Note this reading.
4. Now add 2-3 drops of methyl orange to the same flask the solution turns to yellow or orange.
5. Again titrate this solution with HCl till the color changes from yellow to red. it indicates the methyl orange end point (M). Note this reading.

## OBSERVATION TABLE

**Table 1 : Standardization of HCl**

S. No.	Volume of $\text{Na}_2\text{CO}_3$ in conical flask (ml)	Burette reading Volume of HCl used (ml)		Difference in burette reading (ml)	Concordant reading (ml)
		I.B.R	F.B.R		
1	10.0				
2	10.0				
3	10.0				

**Table 2 : Analysis of alkali mixture**

S. No.	Volume of sample in conical flask (ml)	Burette reading Volume of HCl used (ml)		Concordant reading (ml) (P)	Concordant reading (ml) (M)
		Ph. end point (P)	M O end point (M)		
1	10.0				
2	10.0				
3	10.0				

**CALCULATION****(A) Standardization of HCl**

By Using normality equation:

$$N_1 V_1 = N_2 V_2$$

$$\text{(HCl)} \quad \quad \quad \text{(Na}_2\text{CO}_3)$$

$$N_1 = \text{Normality of HCl (unknown concordant)}$$

$$V_1 = \text{Volume of HCl (Burette reading)}$$

$$N_2 = \text{Normality of Na}_2\text{CO}_3 \text{ solution (N/20)}$$

$$V_2 = \text{Volume of Na}_2\text{CO}_3 \text{ solution (10.0ml)}$$

$$\therefore N_1 = \frac{N_2 V_2}{V_1}$$

**(B) Sample**

The possibility of various ions can be taken by (P) and (M) values of acid used in titration.

(P) = Ph end point / volume of acid consumed in first step.

(M) = Total volume of acid consumed.

$$(M) - 2(M-P) = \text{NaOH}$$

$$(2P-M) = \text{NaOH}$$

$$\text{Eq. Wt. of Na}_2\text{CO}_3 = 53$$

$$\text{Eq. Wt. of NaOH} = 40$$

$$\text{Normality of HCl} = N_1$$

**(C) Calculation of NaOH alkanity**

By using normality equation:

$$N_3 V_3 = N_4 V_4$$

$$\text{(HCl)} \quad \quad \quad \text{NaOH}$$

$$N_3 = \text{Normality of HCl (N}_1\text{)}$$

$$V_3 = \text{Volume of HCl (2P-M)}$$

$$N_4 = \text{Normality of sample solution (unknown)}$$

$$V_4 = \text{Volume of sample solution (25ml)}$$

$$N_4 = \frac{N_3 V_3}{V_4} = \frac{N_1 X (2P - M)}{25}$$

$$\begin{aligned} \text{Strength of NaOH} &= \text{Normality} \times \text{Equivalent weight} \\ &= N_4 \times 40 \text{ gm/lt.} \end{aligned}$$

**(D) Calculation of Na<sub>2</sub>CO<sub>3</sub> alkanity**

$$N_5 V_5 = N_6 V_6$$

$$\text{(HCl)} \quad \quad \quad \text{Sample}$$

$$N_5 = \text{Normality of HCl (N}_1\text{)}$$

$$V_5 = \text{Volume of HCl 2(M-P)}$$

$$N_6 = \text{Normality of sample solution (unknown)}$$

$$V_6 = \text{Volume of sample solution (25ml)}$$



**QUESTIONS**

Q1. What does alkalinity of water indicates?

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Q2. What is the use of determining alkalinity in water?

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Q3. Explain the role of phenolphthalein and methyl orange in determination of various ions causing alkalinity of water?

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Q4. Give various relations of (P) and (M) in the determination of NaOH and  $\text{Na}_2\text{CO}_3$ .

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Q5. Name two indicators used for the determination of alkalinity.

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Q6. Specify pH range of Phenolphthalein and Methyl orange indicator.

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Q7. Give the color change of Phenolphthalein and Methyl orange end point.

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Q8. What is the significance of symbol (P) & (M)?

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Q9. Why do the mixture of (NaOH and  $\text{Na}_2\text{CO}_3$ ) solution is taken to determine the permanent hardness of water sample?

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Q10. Give equivalent weight of NaOH and  $\text{Na}_2\text{CO}_3$ .

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#### 4. Photocell(Detector)

The radiation coming through the filter is allowed to fall on the photocell which measures the intensity of emitted radiations.

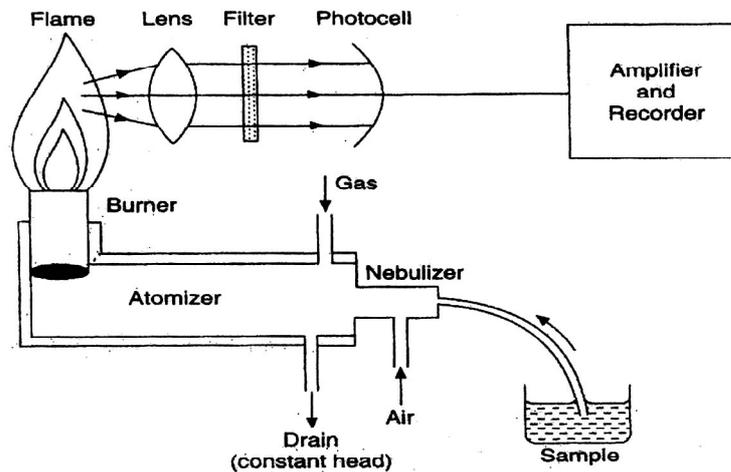
Photocell is used as a detector, because it converts the radiations into an electric current.

#### 5. Amplifier and Recorder

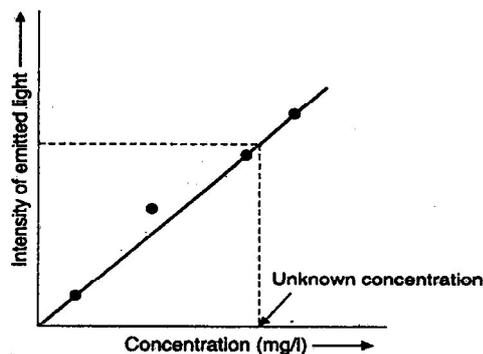
As the current coming out of the detector is weak it is amplified and recorded.

### WORKING OF FLAME PHOTOMETER

Air at given pressure is passed into atomizer. The suction thus produced draws a solution of the sample into the atomizer where it joins the air stream as a fine mist and passes into the burner. Here, the air mixes with the fuel gas supplied to the burner at a given pressure and the mixture is burnt. The resultant radiation from the flame passes through a lens and finally through an optical filters which permits only the radiation characteristic of the element under investigation to pass through the photocell. The output from the photocell is amplified and then recorded (in given figure shown below)



First a calibration curve has to be plotted using solutions of known concentration. This is done by introducing into the flame of known concentrations of the element to be determined, measuring the respective readings and plotting them against the respective concentrations of the standard solutions used. A typical calibration curve of flame photometer is shown as follows.



**PROCEDURE****(A) Preparation of standard solutions**

AR grade sodium chloride is dissolved in one litre of water in a standard measuring flask so that the solution contains the equivalent of one mg of sodium per ml (1 ml of solution has a v e one mg Na). This stock solution is diluted to give four different standard solutions of 10, 5, 2.5, and 1 ppm of sodium ions.

Similarly standard solution for KCl and  $\text{CaCO}_3$

**(B) Construction of calibration curve**

The standard solutions with different concentrations are introduced into the flame and the corresponding digital display readings of emissions intensity are noted by selecting proper filter for Na, K and Ca. When the intensity of the emitted light (digital display reading) is plotted against concentration, a calibration curve is obtained.

**(C) Determination of unknown concentration**

The given sample is then introduced into the flame and the corresponding intensity of the emitted light is found out. The amount of sodium in the water sample can be determined by interpolation.

**OBSERVATION TABLE****Table 1 for Calibration curve**

S.No.	Standard solution of Na/K/Ca(ppm)	Reading of Flame photometer
1		
2		
3		
4		

**CALCULATIONS**

Actual concentrations of Na/K/Ca in the unknown sample can be obtained by converting the reading of flame photometer from the standard curve.

**RESULT**

Na/K/Ca content of given water sample is .....ppm.

**PRECAUTIONS**

1. The glass wares used should be thoroughly washed with distilled water and dried.
2. Proper care should be taken while monitoring the flame.
3. Before and after the use run the flame photometer with distilled water to avoid the chocking in the capability.
4. Prepare standard solution accurately.



## QUESTIONS

Q1. Where do we use Flame photometer?

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Q2. How calibration curve is drawn?

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Q3. Give function of photocell.

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Q4. At which wavelength  $\text{Na}^+$ ,  $\text{K}^+$  and  $\text{Ca}^{++}$  are detected?

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Q5. Give application of flame photometry.

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Q6. Give the source of energy for electron excitation in flame photometry.

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Q7. What are the advantages of flame photometry?

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Q8. How the stock solutions are prepared?

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Q9. Give the principle of flame photometry.

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Q10. On what factor a stable calibration curve depends?

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**Signature of Assistant Professor**

**Signature of C.I. / Principal**

# EXPERIMENT-7

## TURBIDITY MEASUREMENT

### OBJECT

Determination of turbidity in a given sample.

### APPARATUS REQUIRED

Nephelometric turbidimeter, Digital Turbidimeter, sample cells.

### REAGENTS REQUIRED

Hydrazine sulfate  $(\text{NH}_2)_2\text{H}_2\text{SO}_4$ , hexamethylene tetramine  $(\text{CH}_2)_6\text{N}_4$ , water sample under test.

### THEORY

Turbidity is exhibit the optical property that causes scattered and absorbed light rather than transmitting light with no change in direction through the sample. Correlation of turbidity with the weight or particles number concentrations of suspended matter is different because the size shape and refractive index of the particles affect the light-scattering properties of the suspension, Turbidity in water is caused by suspended and colloidal matter such as clay, silt, finely divided organic, inorganic matter and other microscopic organisms.

When it present in significant concentrations, particles consisting of light-absorbing material like activated carbon and dissolved color causing substance cause a negative interference. In low concentrations, these particles tend to have a positive influence because they contribute to turbidity. It may happened in case of some commercial instruments with the capability of either correcting for slight color interference or optically blanking the color effect.

### PRINCIPLE

The Nephelometric method is based on a comparison of the intensity of light scattered by standard reference suspension under the same condition. The higher the intensity of scattered lights, higher the turbidity. Formalizing polymer is used as primary standard reference suspension. It is easy to prepare and is more reproducible in its lights scattering properties than the clay or turbid natural water standards.

The turbidity of a specified concentration of formazin suspension is defined as 4000 NTU.

### Interference

- (i) Turbidity can be determined for any water sample that is free of debris and rapidly setting coarse sediments.
- (ii) Dirty glassware or the presence of air bubbles disturb the surface visibility of the sample will give false results.
- (iii) "True Color" is i.e. water color due to dissolved substance that absorb light, causes measured turbidities to be lowest.
- (iv) This effect usually is not significant in the case of treated water.

### Unit of Measurement

The nephelometric measurements are reported as nephelometric turbidity units (NTU).

## PROCEDURE

### (A) Reagent Preparation

#### (i) Turbidity free water (for setting zero)

Pass distilled water through a membrane filter having a precision pore size of less than 10 microns (Wattman filter no. 42) Rinse collecting flask atleast twice with such filtered water and discard the next 200 ml. Use this filtered water for setting zero of the instrument.

#### (ii) Preparation of stock primary standard formazin suspension

##### (a) Stock Solution-I

Dissolve 1.000g hydrazine sulphate in 100 mL of volumetric flask and distilled water to make upto the mark.

##### (B) Stock solution -II

Dissolve 10.00g hexamine LR grade  $(\text{CH}_2)_6\text{N}_4$ , in mL of volumetric flask and add distilled water to make up to the mark.

(C) In an another 100mL of volumetric flask, mix 5mL of stock solution-I and 5.mL of stock solution-II. Allow it to stand for 24 hours at 35C to 30C to produce in soluble white turbidity corresponding to 4000 NTU.

#### (iii) Preparation of standard formazin (for setting 100 of the intrument)

Dilute 25 mL of the formazin turbidity suspension to 1 litre with turbidity free distilled water to obtain 250 ppm or 1000 NTU for setting 100 of the instrument.

**Note :** Formazin standard 100 NTU should be prepared weekly.

**(B)Standard turbidity suspension:** Dilutions of other values can prepared from this 400 NTU solution by the following dilution method.

Working Solution	1000 NTU std	Distilled Water
400 NTU	40 mL	60mL
200 NTU	20 mL	80mL
100 NTU	10 mL	90mL
10 NTU	1 mL	90mL

**Note:** 10 NTU working solution can be prepared from 100 NTU working solution also. Add 90mL distilled water in 10mL of 100 NTU working solution.

**(C) Calibration of Nephelometer**

- (i) Switch on the instrument and keep it on for sometime.
- (ii) Select appropriate range depending upon expected turbidity of the given sample of water.
- (iii) Set zero of the instrument with turbidity free water using a blank solution and adjust zero (0) with set zero knob.
- (iv) Now in another test tube take standard suspension. Just prepared as in selection for 0 to 200 NTU range, use 100 NTU solution as for higher range use 400 NTU solution as standard.
- (v) Take its measurement and set display to the value of standard suspension with calibrating knob.
- (vi) Now the instrument is ready to take measurement of any solution of unknown turbidity (concentration)

**Note:** For measurement upto 20 NTU kindly use same test tube for reading of blank and standard or sample.

**(D) Measurement of turbidity:**

- (i) Switch on nephelometer turbidimeter and wait for few minutes till it warms up.
- (ii) Set the instrument at 100 on the scale with a 40 NTU standard suspension. In this case, every diversion on the scale will be equal to 0.4 NTU turbidity.
- (iii) Shake thoroughly the sample and keep it for sometime to eliminate the air bubbles.
- (iv) Take sample in Nephelometer sample tube and put the sample in sample chamber and find out the value on the scale.
- (v) Dilute the sample with turbidity free water and again read the turbidity.

Sample No.	Reading	Unit
---	-	NTU

**RESULT**

The turbidity of given water sample -- NTU.

**PRECAUTIONS**

- (i) Sample test tube must be thoroughly cleaned both inside and outside. In case the test tube gets scratched or etched, discard it.
- (ii) Do not touch the test tube where the light strikes i.e. at the sides of the test tube. So hold the test tube only at the top end.
- (iii) Please ensure that the mark on the test tube coincides with the mark on the instrument panel while taking the readings.
- (iv) Fill the test tube with sample or standards which have been thoroughly agitated. Allow sufficient time for the air bubbles to escape otherwise the reading will slowly come down.



## QUESTUONS

Q1. Define turbidity.

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Q2. What are different methods by which turbidity can be reduced?

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Q3. Give full form of NTU.

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Q4. What is nephelometer?

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Q5. How we measure turbidity ?

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Q6. What are different methods by which turbidity can be reduced?

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Q7. Name the methods which are used in determination of suspended materials in natural water and in processing stream?

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Q8. Can we use nephelometric and turbidimetric measurements for other purposes?

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Q9. What is maximum acceptable limit for turbidity.

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Q10. How do you remove the turbidity from water at home?

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# EXPERIMENT-8

## FLASH AND FIRE POINT

### OBJECT

To determine the flash and fire point of lubricating oil by Pensky-Marten's apparatus.

### APPARATUS REQUIRED

Pensky-Marten's Apparatus.

### REAGENTS REQUIRED

Lubricating oil under test.

### THEORY

Flash point is the lowest temperature at which the lubricating oil gives off enough vapors that will ignite for a moment when a test flame is brought near it. Flash point of burning kerosene is 75°F for at least 5 seconds when flame is brought near it. The fire points are 5 to 40°C higher than the flash points.

### Significance of flash and Fire Point

Determination of Flash and Fire point of a lubricating oil is necessary to indicate the fire hazards of petroleum products and evaporation losses under high temperature operations. The knowledge of flash and fire points of lubricating oil is helpful in providing safe guards against fire hazards.

An oil having flash point less than 140°F is called flammable and those with flash points above 140°C are called combustible oil. A good lubricating oil should have higher flash and fire point than its working temperature to ensure safety against fire hazards.

### Flash and Fire Point Apparatus

Flash and fire point of a lubricating oil can be determined with

1. Abel's apparatus
2. Cleveland's open cup apparatus
3. Pensky-Marten's closed cup apparatus

Pensky-Marten's apparatus is the most commonly used apparatus for determination of flash points of oils. This apparatus consists of following features.

#### (i) Oil cup

It consists of a brass cup having 5 cm diameter and 5.5 cm depth and a mark up to which the oil has to be filled.

#### (ii) Lid of the oil cup

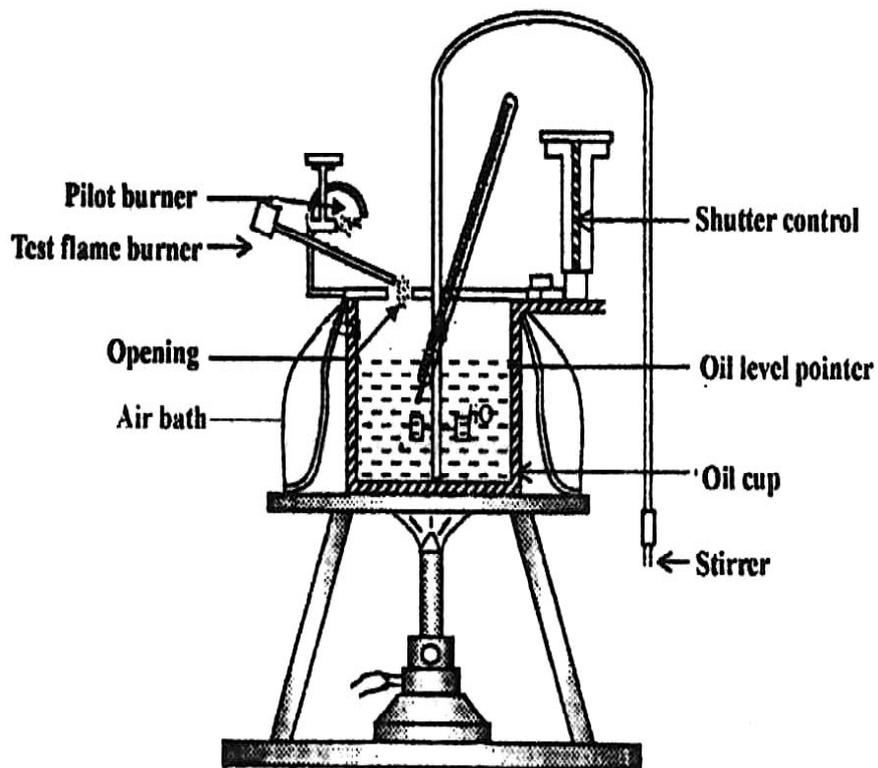
The cover lid of the cup has four openings, one for a special type of stirrer, second for a thermometer, third for an air inlet and fourth a device for introducing the standard flame. At the top of the cup a shutter is provided which has a lever mechanism.

#### (iii) Electrical heater

To heat up the complete assembly in a controlled manner.

## PROCEDURE

1. Clean the oil cup and other parts thoroughly before starting the experiment.
2. The apparatus is kept on a leveled steady table to avoid any fire hazards.
3. Pour the oil to be tested in the oil cup to the level of mark.
4. Cover the lid and insert the thermometer, stirrer and flame exposure device safety in its four openings.
5. Heat the apparatus by using air bath with the help of burner. Air bath and stirrer is used to the apparatus uniformly.
6. The stirrer is rotated at approximately 60 revolutions per minute while heating the apparatus.
7. At every  $1^{\circ}\text{C}$  rise of temperature introduce the test flame of about 4 mm diameter for a moment into oil vapors. This is done by operating the shutter.
8. When a flash appears with weak sound and light, then record the temperature in the thermometer, this temperature is flash point.
9. Allow to cool for sometime, heat again and test the flame for a distinct flash.
10. Further at the test oil at the of  $1^{\circ}\text{C}/\text{min}$ . and test flame as before.
11. The temperature at which the vapors of the oil gives a clear and distinct blue flash for atleast five seconds is recorded as the fire point of the oil.



## RESULT

- (i) The flash point of given oil sample is ..... $^{\circ}\text{C}$ .
- (ii) The fire point of given oil sample is ..... $^{\circ}\text{C}$ .



**QUESTIONS**

Q1. Which factors affect the flash and fire point?

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Q2. What is the flash point of kerosene?

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Q3. How we determine flash and fire point of a lubricating oil?

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Q4. How many openings are there in cup lid?

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Q5. How will you define cloud and pour points?

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Q6. Differentiate flash and fire point.

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Q7. Name any apparatus by which flash and fire points of lubricating oil can be determine along with Pensky-martens apparatus.

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Q8. Give role of air jacket in Pensky-martens apparatus.

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Q9. How can the oiliness of mineral oil be increased?

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Q10. Write main features of Pensky-martens apparatus.

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**Signature of C.I. / Principal**



# EXPERIMENT-9

## VISCOSITY OF A GIVEN SAMPLE OF LUBRICATING OIL

### OBJECT

To determine the viscosity of a given sample of lubricating oil using suitable redwood Viscometer.

### APPARATUS REQUIRED

Redwood viscometer no. 1 and no. 2, stop watch etc.

### REAGENTS REQUIRED

Given lubricating oil.

### THEORY

The viscosity of a lubricating oil can be defined as the tangential force per unit area required to maintain a unit velocity gradient between two adjacent layers in the fluid which are unit distance apart.

Theoretically and academically velocity is generally expressed in centipoise (1/100 poise) or centistoke.

$$F \propto v$$

$$F \propto \frac{1}{d}; F \propto \frac{v}{d}, F = \frac{\eta v}{d}$$

Where  $F =$  Tangential force per unit area

$V =$  Velocity difference between two adjacent layers

$d =$  Distance between two adjacent layers

$\eta =$  Coefficient of viscosity

If  $v =$  1 cm /sec.

$d =$  1 cm

$F =$  1 dyne

Then  $\eta =$  1

The unit viscosity is poise. The viscosity of different lubricating oil can be determined with three types of viscometer namely Redwood, Angler and saybolt viscometer.

Experimental viscosity of an oil can be measured in terms of the (or seconds) taken by an oil to flow through the particular standard apparatus used at particular temperature. Viscosity is also expressed in context with instrument in seconds taken for flow e.g. if the time is 100 seconds at 25°C in Redwood viscometer, then its viscosity is 100 Redwood seconds at 20°C.

Redwood viscometer is available in two sizes.

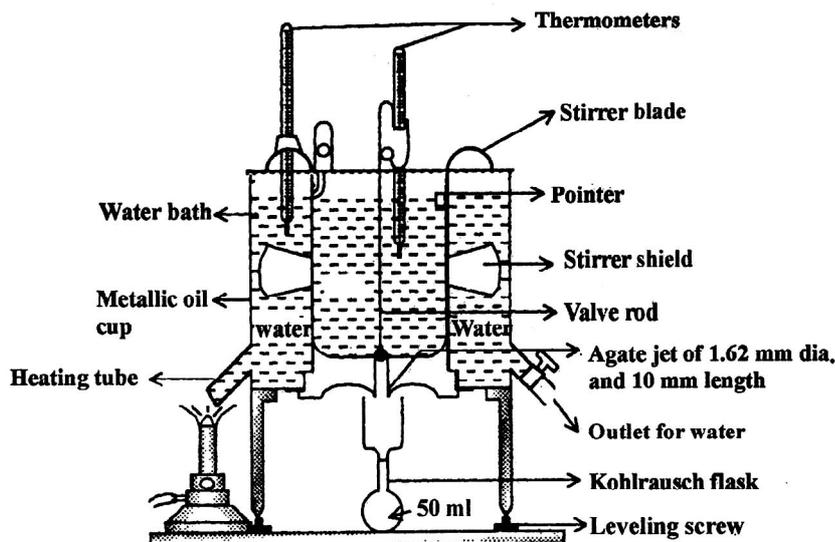
(A) Redwood viscometer no. 1

(B) Redwood viscometer no. 2

Both viscometer are indential in principle, shape and working. They differ in dimensions of the discharge capillary (jet/Orifice) .

Viscometer	Dimensions of Orifice	Length of the Orifice	Useful for the Lubri-cants
AW 1	1.62 mm	10 mm	Lighthouse ( (Kero-sene, Mustard Oil etc.)
RW 2	3.8 mm	50 mm	Highly viscous oils (fuel oil, mobile Oil etc.)

**Construction of Redwood Viscometer No. 1**



**Fig. Redwood viscometer No. 1**

#### (i) Oil Cup

The standard Oil cup which is made up of brass and coated with silver. Height of this is 90 mm and diameter is 46.5 mm. This cup is opened at the upper end. The lid of the cup is fitted with a thermometer to indicated the oil temperature. The bottom of cup is fitted with 10 mm of length and 1.62 mm of diameter. The orifice (jet) is opened or closed by a "valve rod" which is small silver plated ball fixed to a stout wire.

#### (ii) Heating Bath

The cup is surrounded by a cylindrical copper bath containing water with a long side tube on one side for heating water and a tap on the other side for taking to a stout wire.



**QUESTIONS**

Q1. Define Viscosity & Viscosity index.

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Q2 How can we measure Viscosity & Viscosity index of a lubricating oil?

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Q3. Name the viscometers used to measure viscosity of given lubricating oil.

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Q4. What is kohltraush flask?

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Q5. Differentiate between RW1 & RW2?

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Q6. What is the effect of pressure & temperature on viscosity?

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Q7. How does a lubricant work?

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Q8. What is the unit of viscosity ? Give its significance.

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Q9. Give examples of lubricants possessing high viscosity index?

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Q10. Give the exact length of orifice in RW1.

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**Signature of C.I. / Principal**



# **EXPERIMENT-10**

## **CLOUD AND POUR POINT DETERMINATION**

### **OBJECT**

To determine cloud and pour point of given lubricating oil.

### **APPARATUS REQUIRED**

Cloud and pour point apparatus.

### **REAGENTS REQUIRED**

Lubricating oil sample, freezing mixture.

### **THEORY**

The flash and fire point indicate the upper limit of temperature of working conditions for a lubricating oil, whereas the cloud and pour point indicate the lower limit of temperature of working condition for a lubricating oil. When oil is cooled under some specific conditions it first gets cloudy and then solidifies.

#### **Definition of Cloud Point**

The particular temperature at which lubricating oil becomes cloudy or hazy in appearance is called cloud points .

#### **Definition of Pour Point**

The particular temperature at which the oil becomes semi solid and ceases to flow. This temperature is known as pour point.

#### **Significance of cloud and pour point**

Cloud and pour point indicates the suitability of lubricants in cold condition. Lubricant used in a machine working at low temperature should possess low pour point otherwise solidification will cause jamming of the machine.

#### **Construction of apparatus**

The apparatus used for the determination of cloud and pour consist of the following parts.

##### **(i) Flat bottomed tube**

It is a glass tube with standard dimensions i.e. 12 cm height and 3 cm diameter.

##### **(ii) Glass or metal jacket**

The tube is enclosed in a glass or metal jacket which is filled with freezing mixture and a thermometer.

### **PROCEDURE**

1. Clean and dry the flat bottomed tube.
2. Pour the oil inside the tube and close it with a cork.
3. Place the thermometer inside the oil properly and in the cooling bath to note the temperature.
4. The flat bottomed tube is then kept in the freezing mixture. The freezing mixture can be prepared as follows.



**QUESTIONS**

Q1. What are the main parts of cloud and pour point apparatus?

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Q2. What should be done to improve the cold temperature performance of bio diesel.

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Q3. Name the substance which can be added to reduce cloud and pour point.

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Q4. Give diameter and height of flat bottom tube.

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Q5. How can we decrease the cloud and pour point of fuel?

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Q6. Name the apparatus by which cloud and pour point can be determined?

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Q7. How cloud and pour point affect the efficiency of lubricating oil?

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Q8. Name the freezing mixture which is generally used in cloud and pour point apparatus?

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Q9. Machines working at low temperatures should possess low or high pour point?

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Q10. Why lubricants show cloud and pour point?

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**Signature of Assistant Professor**

**Signature of C.I. / Principal**