

# INDIAN INSTITUTE OF TECHNOLOGY

DATE 11.08.2022

Determination of ASTM Distillation Characteristics  
of Gasoline

SHEET NO. 1

AIM:- Determination of distillation characteristics (boiling, volatility) of petroleum product using ASTM methods (D-86). The boiling range gives information on the composition, properties and the behaviour of fuel storage and use.

## THEORY:-

- Charge Volume :- n - the volume of specimen, 100ml, charged to the distillation flask at specified temperature.
- Decomposition :- n - of a hydrocarbon, the pyrolysis or cracking of a molecule yielding smaller molecules with lower boiling points than the original molecule.
- Decomposition point :- n - the corrected thermometer reading that coincides with the first indication of thermal decomposition of the liquid in the flask.
- Dry Point :- n - the corrected thermometer reading that is observed at the instant the last drop of liquid evaporated from the lowest point in the distillation flask.
- Dynamic Holdup :- n - the amount of material present in the neck of the flask, in the side-arm of the flask and in the conductor tube during the distillation.

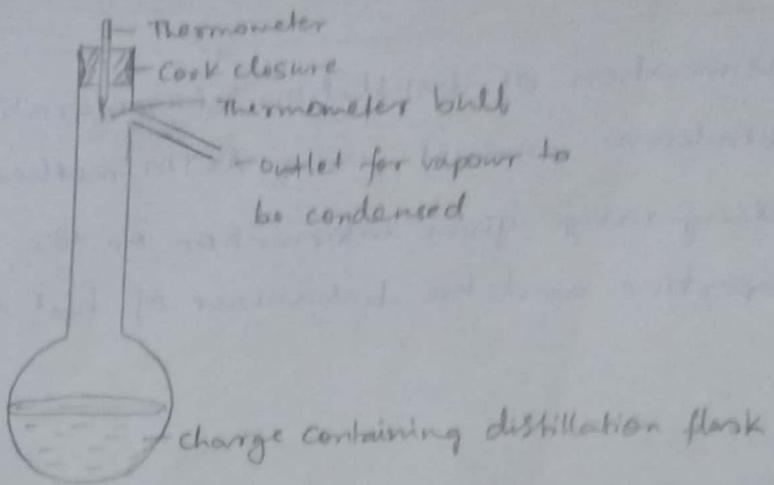


Fig. 1

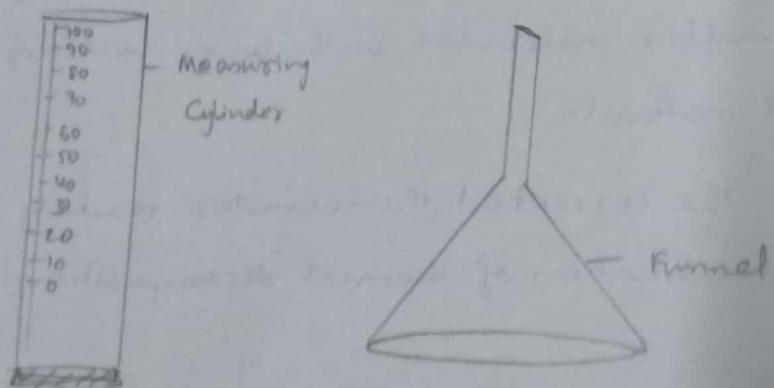


Fig. 2

Fig. 3

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SEMESTER 2

- Emergent steam effect :- n- the distillation effect in temperature reading caused by the use of total immersion mercury in glass thermometer in the partial immersion mode.
- End point :- n- the maximum corrected thermometer reading obtained during the test. This is also called the final boiling point.
- Initial boiling point :- n- the corrected thermometer reading that is observed at the instant the first drop of condensate falls from the lower end of the condenser tube.

## APPARATUS :-

- i) Distillation flask
- ii) Thermometer
- iii) Electric heater
- iv) Water bath
- v) Collection cylinder and measuring cylinder.
- vi) Funnel

## PROCEDURE :-

A 100ml specimen of the sample was distilled under the prescribed condition, systematic observations of temperature readings and volumes of condensate were made. The volume of the residue and the losses are also found. The temperature reading that was observed at the instant of the first drop of condensate falling from the lowerend of the condenser tube was noted.



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This temperature is known as Initial Boiling Temperature (I.B.P.). The volume of the condensate obtained in the receiving cylinder expressed as a percentage of the change in volume is called "percent recovered distillate". The maximum thermometer reading obtained during the test is called "Final Boiling Point" (F.B.P.)

## OBSERVATION :-

Initial Boiling Point (I.B.P) =  $47^{\circ}\text{C}$

Volume of the collected lig. (%)

0%

10%

20%

30%

40%

50%

60%

70%

80%

90%

93%

Temperature ( $^{\circ}\text{C}$ )

$47^{\circ}\text{C}$

$55^{\circ}\text{C}$

$61^{\circ}\text{C}$

$68^{\circ}\text{C}$

$99^{\circ}\text{C}$

$113^{\circ}\text{C}$

$127^{\circ}\text{C}$

$141^{\circ}\text{C}$

$156^{\circ}\text{C}$

$178^{\circ}\text{C}$

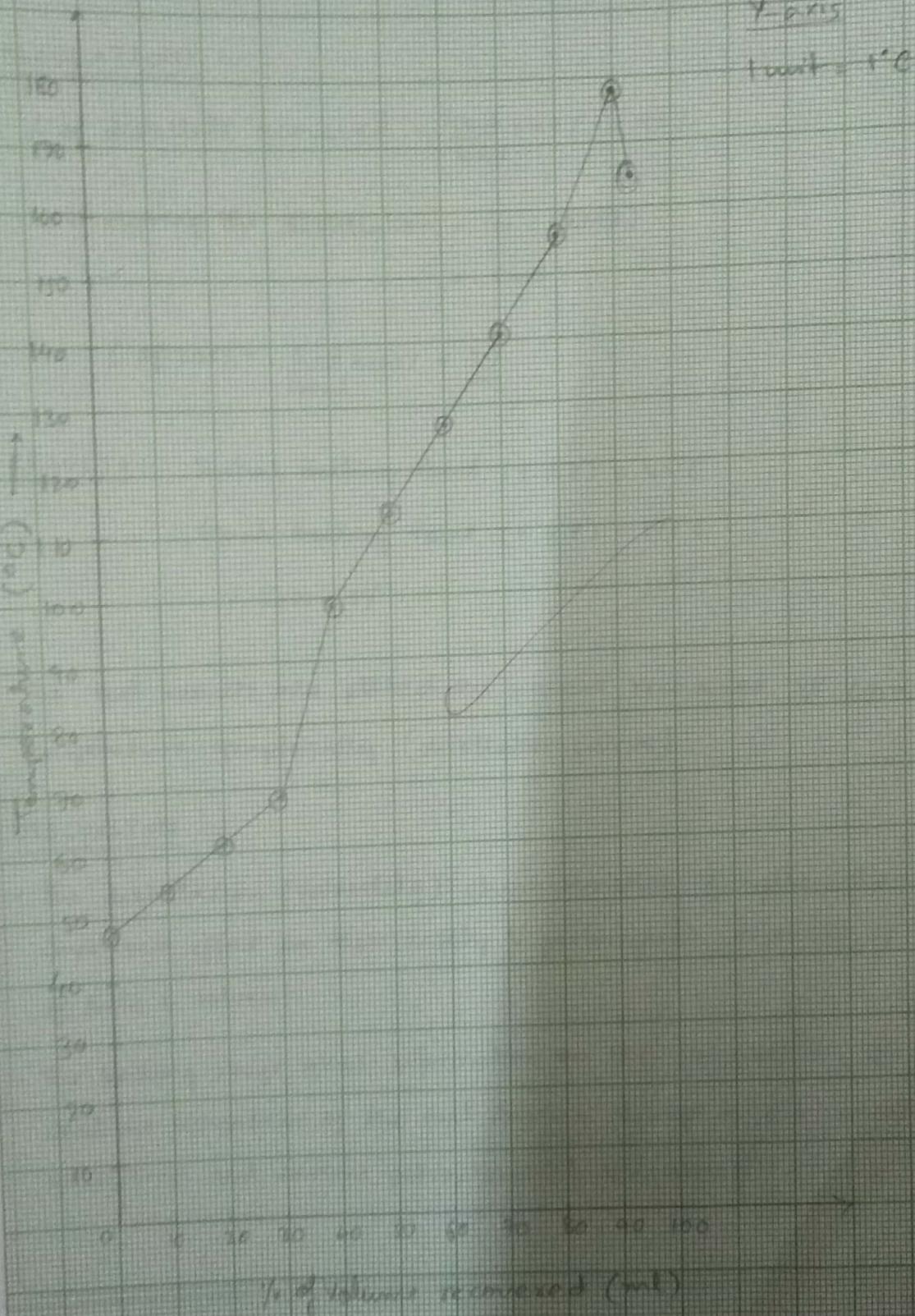
$165^{\circ}\text{C}$

Final Boiling Point (F.B.P) =  $178^{\circ}\text{C}$

Dry Point =  $165^{\circ}\text{C}$

Volume of recovered distillate = 93ml

Plot of Temp (°C) vs volume(%) distilled



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- Volume of residue left = 2ml

## CALCULATIONS :-

$$\% \text{ of distillate recovered} = \frac{93 \text{ ml}}{100 \text{ ml}} \times 100\% = 93\%$$

$$\begin{aligned}\text{Loss during the distillation} &= 100 - (93+2) \\ &= 5 \text{ ml}\end{aligned}$$

$$\% \text{ of Loss} = \frac{5 \text{ ml}}{100 \text{ ml}} \times 100\% = \underline{\underline{5\%}}$$

RESULTS :- Initial Boiling Point (I.B.P) = 47°C

Final Boiling Point (F.B.P) = 178°C

$$\% \text{ Loss} = 5\%$$

The test results are plotted on percent recovered v/s corresponding temperature graphically (distillation curve).

## DISCUSSION :-

- The distillation (volatility) characteristics of hydrocarbons have an important effect on their safety & performance, especially in the case of fuels & solvents.
- The boiling range gives information on the composition, the properties and the behaviour of the fuel during storage in the industries like HPCL, ONGC and various petrochemical industries.

- Volatility as it effects rate of evaporation, is an important factor in the application of many solvents, particularly those used in paints.
- Distillation limits are often included in petroleum product specifications in commercial contract arguments, process refinery / control applications and for compliance to regulatory rules.
- Characteristic indications of thermal decomposition are evolution of fumes or erratic temperature readings that usually decrease after any attempt is made to adjust the heat.
- In the course of experiment, we ignored the fact that ambient pressure isn't equal to 1 atm or 101.3 KPa. But the standard condition mentioned in D-86 method of ASTM incorporates the pressure to be 101.3 KPa as standard. Thus, there should be some correction to be considered in results.
- ASTM-D Standards originally known as the American Society for Testing and Materials is an international standards organisation that develops and publishes voluntary consensus technical standards for a wide range of materials, products, systems & services.

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Exp-2(a): Determination of Reid Vapour pressure of a gasoline.

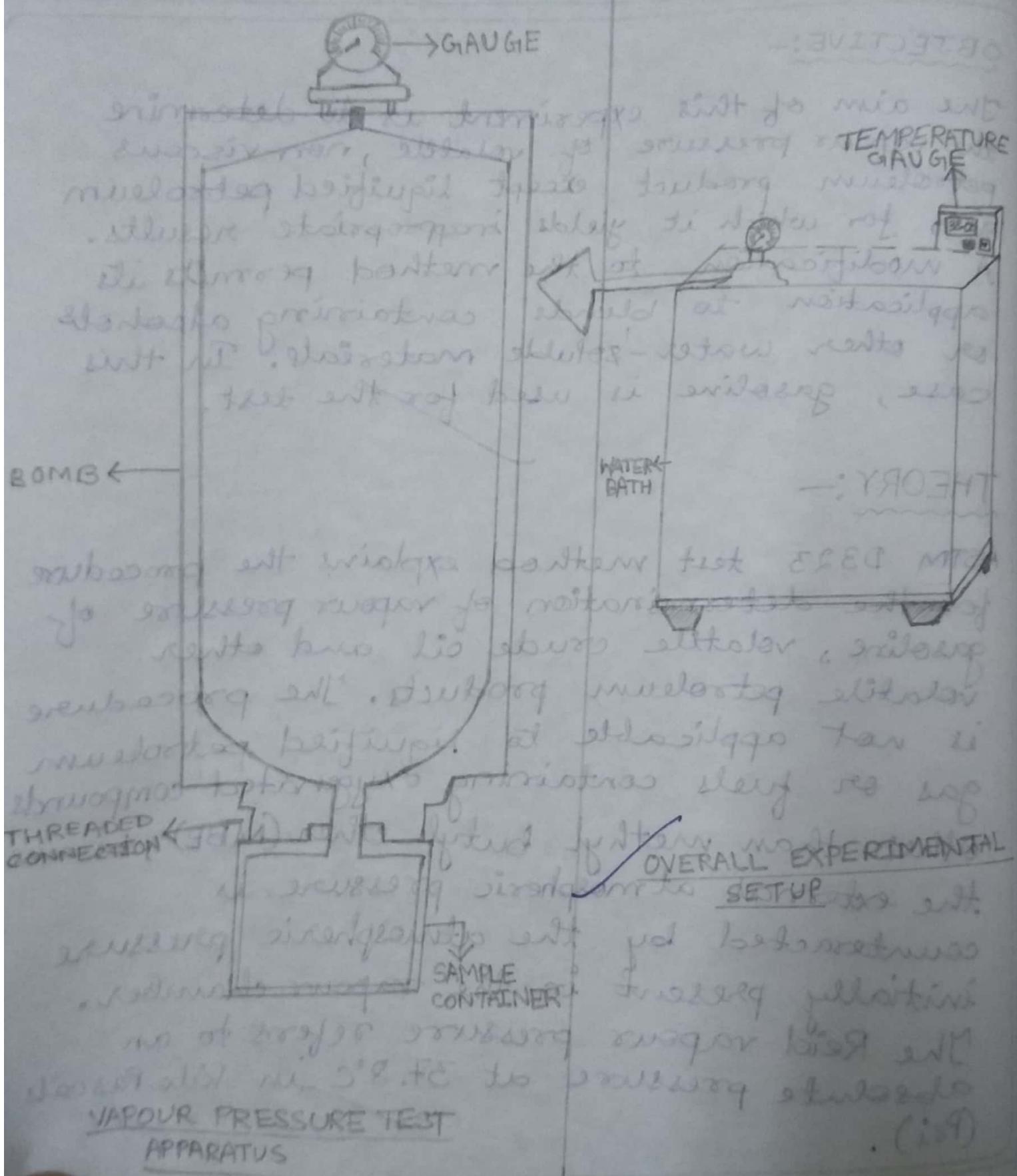
SHEET NO. 6

## OBJECTIVE:-

The aim of this experiment is to determine the vapour pressure of volatile, non-viscous petroleum product except liquified petroleum gas, for which it yields inappropriate results. A modification to the method permits its application to blends containing alcohols or other water-soluble materials. In this case, gasoline is used for the test.

## THEORY:-

ASTM D323 test method explains the procedure for the determination of vapour pressure of gasoline, volatile crude oil and other volatile petroleum products. The procedure is not applicable to liquified petroleum gas or fuels containing oxygenated compounds other than methyl butyl ether (MBE), because the external atmospheric pressure is counteracted by the atmospheric pressure initially present in the vapour chamber. The Reid vapour pressure refers to an absolute pressure at 37.8°C in kilo Pascals (Psi).



## APPARATUS REQUIRED:-

- i) Gasoline-chamber (vapour-pressure bomb).
- ii) Air chamber (a cylindrical vessel on the upper part of the vapour pressure bomb).
- iii) A Bourdon-type of spring gauge.
- iv) Water bath (usually maintained at  $38.0^{\circ}\text{C} \pm 0.1^{\circ}\text{C}$ )

## PROCEDURE:-

- i) The liquid sample container was thoroughly cleaned and gas sample was filled in it up to the brim.
- ii) The water bath was heated and maintained at around  $30^{\circ}\text{C}$ .
- iii) Both the Reid Vapour pressure apparatuses are shaken constantly to eliminate the presence of liquid bubbles in it.

## OBSERVATION:-

A pressure of 6.5 Psi or  $0.47 \text{ kg/cm}^2$  was observed on both the Reid Vapour pressure apparatuses when immersed in the water bath for 15 minutes while the water bath was

maintained at  $38.1^{\circ}\text{C}$ .

### RESULTS:-

Reid Vapour Pressure of the gasoline sample was found to be 7 Psi.

### DISCUSSION:-

- i) Reid Vapour pressure is the absolute vapour pressure of a volatile fuel at  $37.8^{\circ}\text{C}$  ( $100^{\circ}\text{F}$ ) as was evaluated using ASTM method D323A.
- ii) The term 'dry vapour pressure equivalent' is often used in place of Reid Vapour pressure. The values of dry vapour pressure equivalent and Reid Vapour pressure of a gas sample are close but not identical.
- iii) Reid Vapour pressure (RVP) is the most commonly used metric in discussing automatic gasoline vapour pressure and its regulation.
- iv) RVP is controlled to reduce vapour lock concerns in fuel systems where they still exist.
- v) Most of the RVP is provided by isomers of propane, butane and pentane. Butane is blended in various proportions to adjust RVP.

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- vi) High RVP compounds are needed for vehicle starting and warm up of spark engines. If the RVP is too low, then there won't be enough hydrocarbon vapor available to start a cold engine. ✓

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Exp 2(b) : Determination of Flash Point of an Oil  
by Pensky - Martens Apparatus

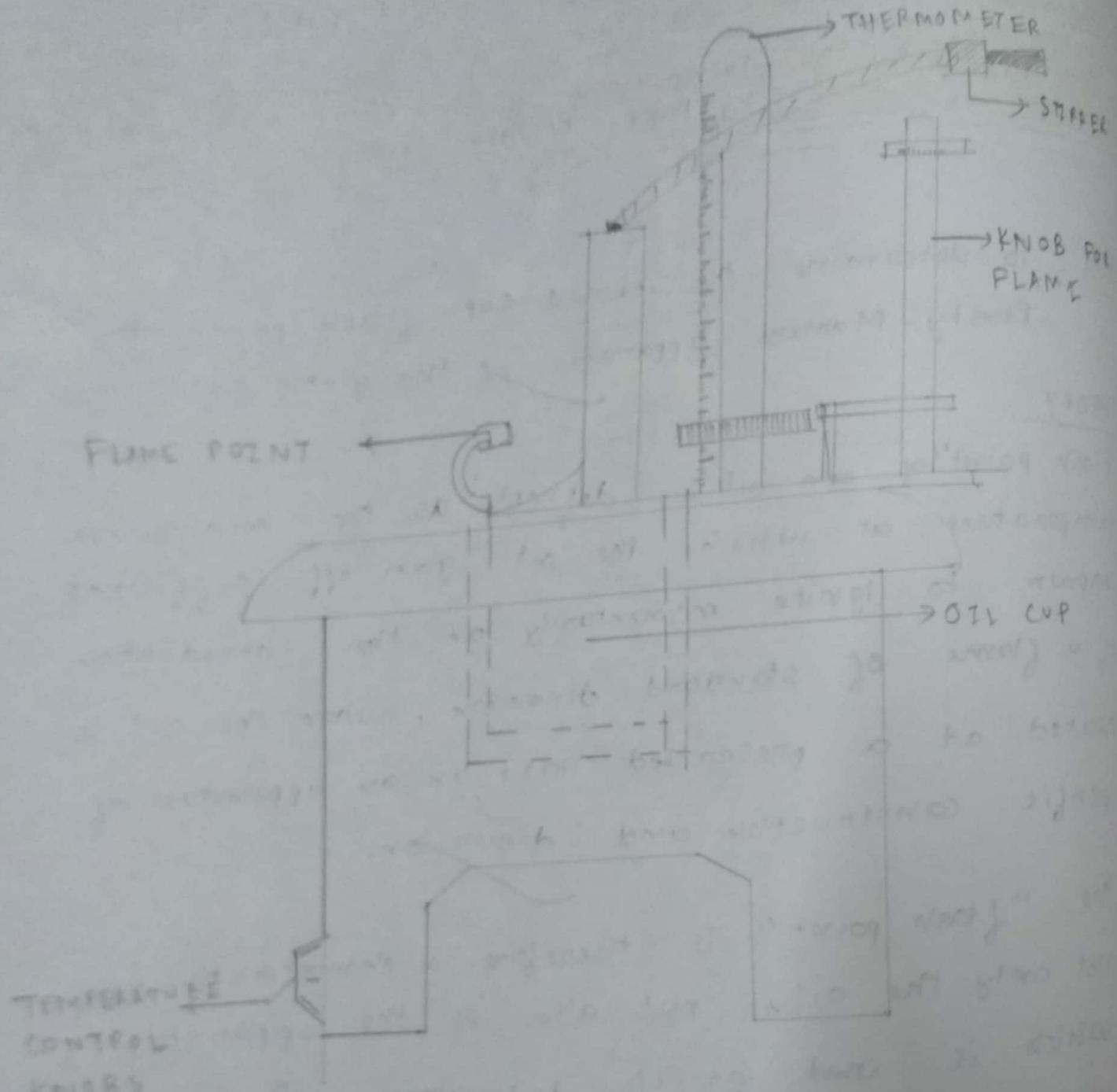
SHEET NO. 10

AIM : To determine the closed-cup flash point by  
Pensky - Martens apparatus of the given fuel

## THEORY:

"Flash point" of an oil is defined as the minimum temperature at which the oil gives off sufficient vapour to ignite momentarily on the introduction of a flame of standard dimension, when the oil is heated at a prescribed rate in an apparatus of specific construction and dimensions.

The "flash point" is therefore a characteristic of not only the oil, but also of the apparatus which is used for its determination. The apparatus may or may not be provided with a lid and "flash-point" obtained is correspondingly called "closed" or "Open". The "closed" "flash-point" of any oil is always lower than "Open" for obvious reasons. There are two forms of standard apparatus specified by the Institute of Petroleum (U.K.) one is Abel apparatus, designed for determining "Flash Point" up to



▲ Penetrometer-Mantle Apparatus

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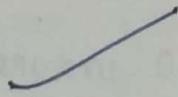
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SHEET NO. 11

120 F, and the other Pensky - Martens apparatus, for determining "flash point" above 120 F.

## APPARATUS :

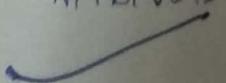
- (i) Pensky - Martens closed cup arrangement,
- (ii) Bunsen Burner
- (iii) Stirrer
- (iv) Thermometer



## PROCEDURE :

The oil cup is cleaned, dried and filled in upto the given mark. The lid is placed in position, the thermometer is inserted in the thermometer holder and the cup is put in position. The test flame is adjusted to the size of a bid, 1mm in diameter.

Heating is started and continued at a rate of 9 to 11 F per minute and stirrer is rotated at an rpm of 60 to 120. Starting at a temperature of atleast 30 F below the "flash-point" the test flame is applied at temperature intervals of 2F. Stirring



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SHEET NO. 12

should be stopped when the test flame is applied.  
The temperature at which a distinct flash is visible  
in the two observation point is recorded.

→ Observation:

Material taken : Kerosene Oil

Barometric Pressure : 760 mm Hg

Temperature ( $^{\circ}\text{C}$ )	Observation
41 $^{\circ}\text{C}$	No
42 $^{\circ}\text{C}$	No
43 $^{\circ}\text{C}$	No
44 $^{\circ}\text{C}$	No
:	
53 $^{\circ}\text{C}$	Flash Point Observed

RESULT :

Flash Point of a given sample (Kerosene) = 53  $^{\circ}\text{C}$

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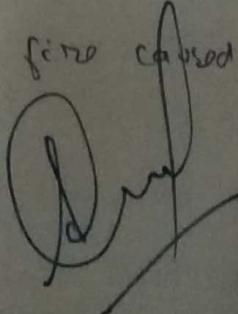
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SHEET NO. 13

## DISCUSSION

- i. Flash point is the lowest temperature at which there will be enough flammable vapour to induce ignition when an ignition source is applied.
- ii. Flash point is a characteristic used to distinguish between flammable and combustible fuels, and is as such used to characterise the fire hazards of fuels.
- iii. Flash points are measured by heating a liquid to specific temperatures under controlled conditions and then applying a flame, in either "open cup" or "closed cup" apparatus depending on conditions desired to be mimicked from industry point of view.
- iv. The two standardised apparatus for determining the flash point are Abels apparatus, which is open cup type, and Pensty Martens apparatus, which is closed cup type.

- (v) The apparatus is equipped with a stirrer whose function is to homogenise the sample under observation to avoid discrepancies.
- (vi) A possible source of error in observation may come from the fact that some amount of vapour of the fuel may escape when the opening is observed against the test flame.
- (vii) Measured pressure may also vary from the required pressure. It is to be taken care of that the opening must be unlatched for a very short amount of time to prevent escape of gas, along with regular stirring.
- (viii) Flash point is different from a related term, fire point, in that, the vapour need not necessarily sustain the fire caused by the test flame.



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DATE 1-09-22

Experiment 3 : Determination of Gum content (existent) in petrol sample

SHEET NO. 15

AIM :- To determine the gum content (existent) in a given petrol sample.

THEORY :- This test method covers the determination of the existent gum content of aviation fuel, and the gum content of motor gasolines or other volatile distillates in their finished form, (including those containing alcohol and other type oxygenates and deposit control additives) at the time of test.

Existent gum :- The evaporation residue of aviation fuels, without any further treatment, under standard conditions of test.

APPARATUS :- (i) Evaporation bath,  
(ii) Glass Beaker  
(iii) Balance

PROCEDURE :- Wash the beaker with the solvent until free from gums and dry in oven. Weigh the test beakers to the nearest 0.1 mg and record the weigh. Using a measuring cylinder measure 50 ml of gasoline



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SHEET NO. 16

sample into each breaker and place the filled breaker in the evaporation chambers. Evaporate the sample by means of air using conical jets. Center the jets above the surface of the liquid. Maintain the temperature and the rate of flow and allow the evaporation to proceed for 30 minutes. At the end of the heating period, cool the breakers weigh the breakers and record their weights. Determine the existing gum content in mg/100 ml.

### OBSERVATIONS :-

S.No.	Wt of empty breaker	Wt of breaker + Petroleum	Wt of breaker after evaporation (with gum content)
1.)	55.695	59.057	55.699
2.)	60.103	63.588	60.107
3.)	56.913	60.323	56.917
4.)	54.180	57.563	54.184
5.)	54.910	58.405	54.923

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Specific Gravity of bottle :-

$$\text{Total wt of } \frac{\text{all}}{\text{empty}} \text{ beaker} = 281.801 \text{ g}$$

$$\text{Total wt of } \frac{\text{all}}{\text{beaker with petroleum}} = 298.936 \text{ g}$$

$$\text{Density} = \frac{(298.936 - 281.801) \text{ g}}{25 \text{ ml}} = \frac{17.135}{25}$$

$$= 0.6854 \text{ g/ml}$$

Calculation :-

For data set 1:-

$$55.695$$

$$M_{\text{empty}} = \cancel{55.684} \text{ g}$$

$$M_{\text{beaker + petrol}} = 59.057 \text{ g}$$

$$M_{\text{beaker after evaporation}} = 55.699 \text{ g}$$

$$\text{Existent gum content of beaker 1} = 55.699 - 55.695 \text{ g}$$

$$= 0.004 \text{ g}$$

$$\text{Volume of petrol} = 59.057 - 55.695 = \frac{3.362}{0.6854}$$

$$= 4.9054 \text{ ml}$$

$$\text{Gum content / 100 ml sample} = \frac{0.004 \times 100}{4.9054} = 0.08154 \text{ g}$$

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SHEET NO. 10

Sno.	mass of gasoline (m)	volume of gasoline = $\frac{m}{\rho}$ (ml)	gum (g/ml)	gum content per 100 ml
1.)	3.362 g	4.9054 ml	0.0049	0.081549
2.)	3.485 g	5.0846 ml	0.0049	0.078669
3.)	3.410 g	4.9751 ml	0.0049	0.080409
4.)	3.383 g	4.9358 ml	0.0049	0.081049
5.)	3.495 g	5.0992 ml	0.0039	0.09849

Average Gum Content = 
$$0.8154 + 0.07866 + 0.08040 + 0.08104 + 0.09849$$

$$\sqrt{= 0.080016 \text{ g}/100\text{ml sample}}$$

Result : Existent gum content in given Sample

$$= 0.029 \text{ g}/100\text{ml of sample}$$

Calcd. gum (Ans)

Longer time the remaining part remains  
unchanged and present gum formulae undergoes  
oxidation and having addition. Even  
additions added to your entire sample  
is reduced, mostly available in the market  
other fruits can be added to prevent  
oxidation.

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## DISCUSSION :-

Gasoline is a volatile mixture of hydrocarbons that is used in spark-ignition engines. It is a complex mixture composed of olefins, paraffin, naphthenic, and aromatic hydrocarbons ( $C_4 - C_{12}$ ), among other substances in a smaller concentrations. In several countries, such as Brazil, ethanol is used as a renewable fuel for SI engine. During storage some classes of hydrocarbons in gasoline blends particularly olefins and diolefins, are able to slowly react, at ambient temperatures, with oxygen in air. The formed oxidation products are responsible for the formation of an insoluble solid commonly called gums / deposits.

Consequently it is necessary to predict gasoline blend behaviour and prevent gum formation, improving gasoline quality and using additives. Even if the numbers of publications related to gum content formation in gasoline is reduced, results available in the literature for other fuels can be applied to gasoline issue.

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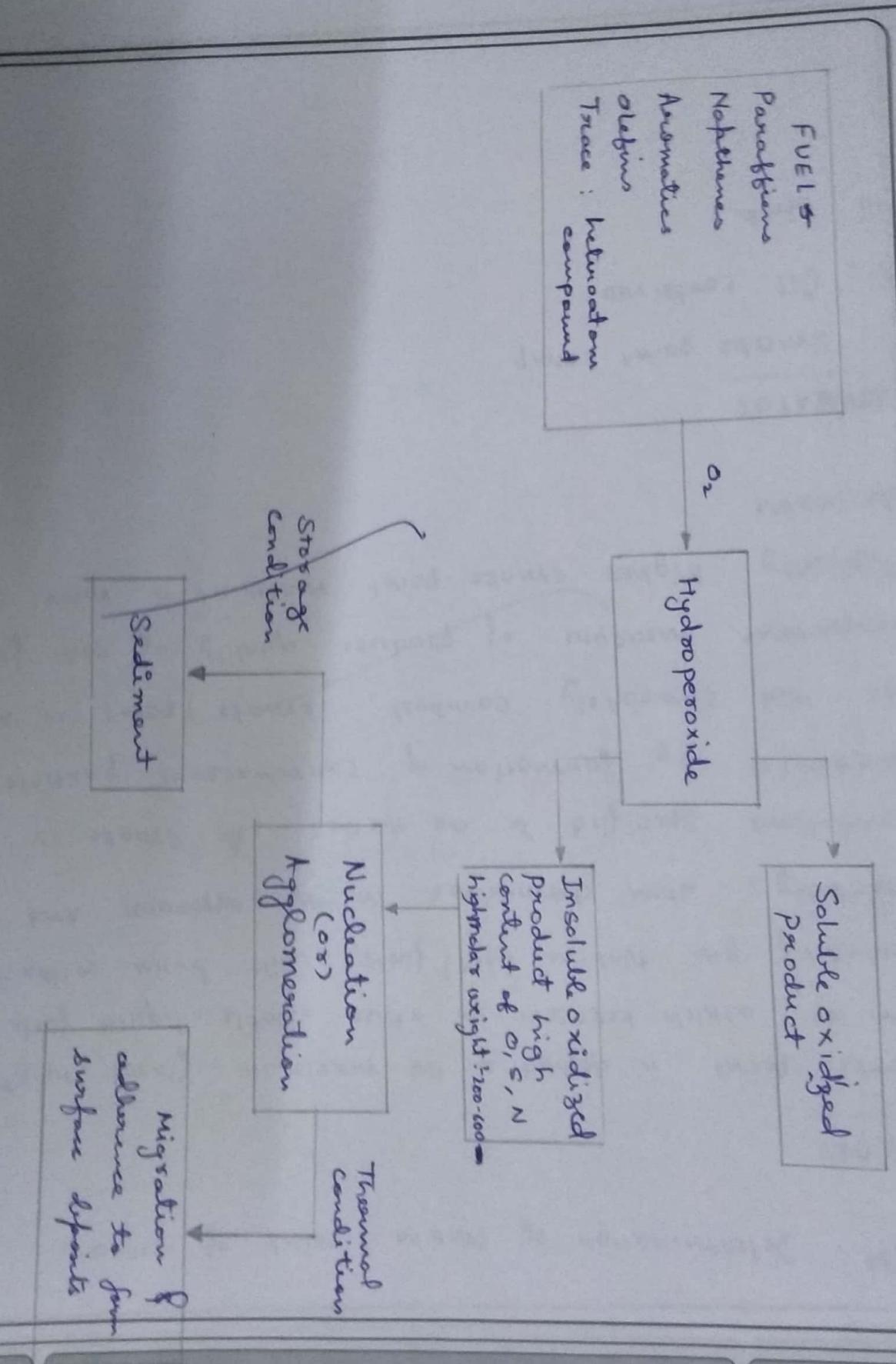
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The gum content can be used to judge the quality of various products. The amount of gum indicates the condition of the sample at the time of measurement. Large quantities of gum are an indicator for the contamination of fuel caused by higher boiling oils or particles. Since the gum content can change according to the age of the fuel and the exposure to the oxygen, the reported content is only true at the time of measurement.

Generally the gum content reflects inappropriate production process and poor fuel handling in the refinery or the storage facility. The amount of gum should be as low as possible since the use of fuel with high gum contents can lead to deposits in induction systems or cause intake valve and fuel injections to stick.

According to the fuel specifications, ASTM D 1655 and ASTM D 4814 the limitations for the gum content are 7 mg per 100 ml and 5 mg/100 ml for motor gasoline.

Fig :- Formation of gum



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DEPT. OF EXP. & IND. PROCESSES

SHEET NO. 22

## AIM: Determination of smoke point of benzene

### THEORY:

Smoke point is defined as the maximum flame height at which benzene or other volatile liquid fuel including gas turbine (jet) fuel will burn without smoking, when determined in the apparatus and under conditions specified in the method. As smoke is produced by combustion of carbonaceous particles that do not completely combust, smoke point is an important measure of product quality of such fuels. Typically higher smoke point material is more desirable.

### APPARATUS:

- i. Smoke point lamp
- ii. Oil container
- iii. Wick

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SHEET NO 23

## SIGNIFICANCE AND USE

The smoke point of any grade of kerosene is an important determinant of the effect of the fuel on the engine as well as the environment.

## PROCEDURE

20 ml of kerosene was introduced in a clean dry oil container. A piece of wick is soaked in the sample under test and properly placed in the wick holder and the wick holder is placed in the container. The wick is horizontally cut and trimmed free of frayed ends, so that 6 mm of wick project from the container. Now the oil container is inserted into the lamp and the wick is lit. The wick is adjusted so that the flame is about ~~ten cm~~ height and the lamp is allowed to burn for some time. The wick is raised until a sooty flame appears. Then the wick is slowly lowered until a well rounded tip without any smoke is obtained, by properly adjusting flame height. Then the height of the flame is estimated.

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to the nearest mm, and recorded as the smoke point. This experiment is repeated for 4 times, and the mean of the determination to the nearest 1 mm is reported as the smoke point of the sample.

### OBSERVATIONS :

Sl. No.	Flame height of smoke point (in mm)
1.	22 mm
2.	24 mm
3.	23 mm
4.	21 mm

### CALCULATION :

Average flame height at smoke point =  $\frac{1}{4} \times (22\text{mm} + 24\text{mm} + 23\text{mm} + 21\text{mm})$   
 =  $\frac{90}{4}\text{mm}$   
 = 22.5 mm  
 $\sim 23\text{mm}$  (nearest integer)

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## RESULT :

Flame height at the smoke point for given sample is 23mm.

## DISCUSSION

- i. Aromatics or naphthalenes enhance low smoke point, and hence, smoke point also gives a measure of these components in a fuel sample.
- ii. A fuel that is high in soot/smoke production may lead to excessive carbon build up in turbine engines, causing failure. Thus, a high smoke point for the fuel is usually desirable.
- iii. Smoke point tends to normalize using various methods, the most widely used being threshold sooting index (TSI).
- iv. To improve smoke point, a method called hydro treating is used, where aromatic rings leading to the soot are saturated with additional compounds.

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- v. Enron may set in due to mis placing of wick on wick holder ~~inside the chamber~~, or also due to ~~effect~~ of clumsy endings of wicks.
- vi. Care must also be taken to analyse the flame height appropriately and perpendicularly.
- vii. The standard method for checking smoke point is ~~ASTM D1332~~.
- viii. Replacing low smoke point grades with higher ones is also advantageous from an environmental perspective, as soot leads to damages like haze and acidification of lakes and rivers, which are detrimental to the environment.

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DATE 08/09/2022

Experiment 4(6):  
Determination of Conradson Carbon Residue

SHEET NO. 27

## OBJECTIVE :-

To determine conradson carbon residue of a heavy oil sample.

## APPARATUS :-

- Sand bath Iron crucible
- Hood
- Burner

## THEORY :-

This test method covers the determination of the amount of carbon residue left after evaporation and pyrolysis of an oil and is intended to provide some indication of relative coke forming properties. This test method is generally applicable to relatively non-volatile petroleum product which partially decompose on distillation at atmospheric pressure.

Petroleum products containing ash forming constituents as determined by the test method will have an erroneously high carbon residue depending upon the amount of ash formed.

## PROCEDURE :-

- Oil sample was weighed and taken in a crucible by a mass of ~5gm
- The crucible was placed inside sand bath and covered by a chimney and hood.

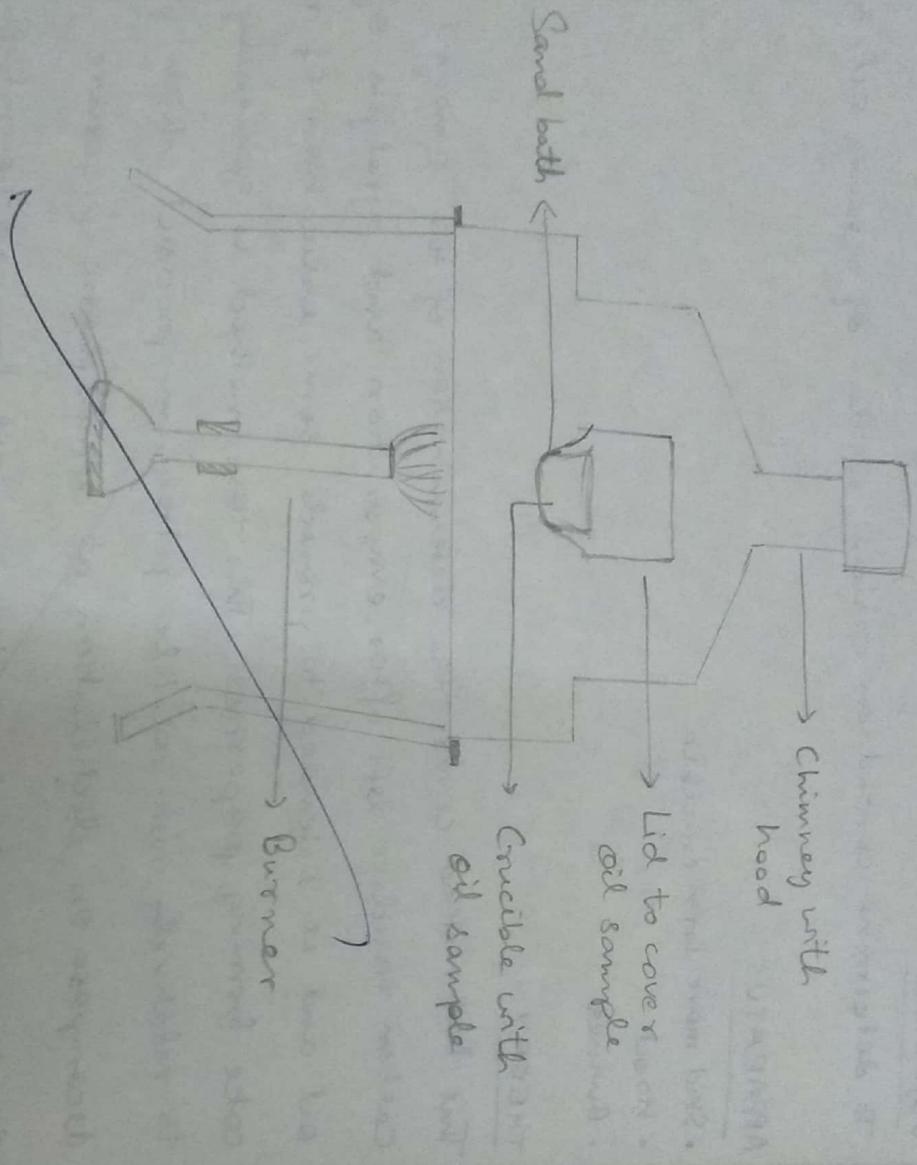


Fig. - Apparatus for Conradson Carbon Residue determination

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(iii) The entire setup was placed on a suitable stand.

(iv) The setup was strongly heated for 30 minutes - 1 hour with the help of gas burner.

## SIGNIFICANCE AND USE :-

The carbon residue value of burner fuel serves as a rough approximation of the tendency of the fuel to form deposits in vaporizing pot-type and sleeve type burners.

The carbon residue value of motor oil, while at one time is regarded as indicative of the amount of carbonaceous deposits a motor oil would form in the combustion chamber of an engine.

## OBSERVATION :-

- weight of empty crucible = 68.403 gm
- weight of heavy oil = 4.844 gm
- weight of crucible + heavy oil = 73.247 gm
- final weight of crucible + residue = 68.815 gm
- weight of residue =  $(68.815 - 68.403)$  gm  
= 0.412 gm

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## CALCULATION :-

$$\text{Percentage (\%)} \text{ weight of residue} = \frac{0.412 \text{ gm}}{4.844 \text{ gm}} \times 100$$

$$= 8.5\% \quad \text{Very good}$$

## RESULT :-

Conradson Carbon Residue in the heavy oil sample is 8.5%.

## DISCUSSION :-

Quantitatively, the test measures the carbonaceous residue remaining after the oil's evaporation and pyrolysis.

The crucible has to be cleaned ~~properly~~ before performing the experiment because some ~~residues~~ might be present previously. It is an ~~approximate~~ measure of the carbon-residue as some other components were also present in insignificant amount.

Conradson Carbon Residue conversion is a critically important reaction in residue hydroprocessing

The H-2495, CCR Apparatus is used to determine the amount of carbon residue.

We have heated the crucible for 1 hour and then the evaporation of other components stopped which indicate only the residue

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is left. It is expressed as a percent of the whole fuel. Fuels with high CCR values have an increasing tendency to form carbon residue on injection nozzles, pistons and in the ports of two stroke engines.

Efficiency & performance of these components are expected to decrease due to the wear & tear & deposition of carbon.

Dekhar  
K.S. 1/2022

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Experiment 5(a): Determination of  
Aniline point of an oil sample

SHEET NO. 31

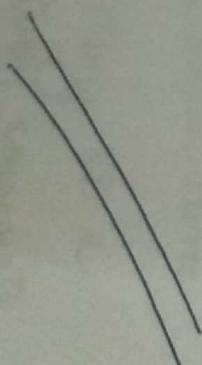
Aim:- Determination of aniline point of diesel

Terminology:- For the purpose of this method,  
the following definition shall apply.

Aniline point of an oil sample is the minimum  
equilibrium temperature at which equal volume  
of oil is completely miscible in equal volume of  
aniline.

## APPARATUS :-

- (1) U shaped Test tube
- (2) Water jacket
- (3) heating arrangement
- (4) stirrer



Theory:- Specified volume of aniline and sample  
are placed in a U-tube and mixed mechanically.  
The mixture is heated at controlled rate until  
two components are completely miscible. The mixture

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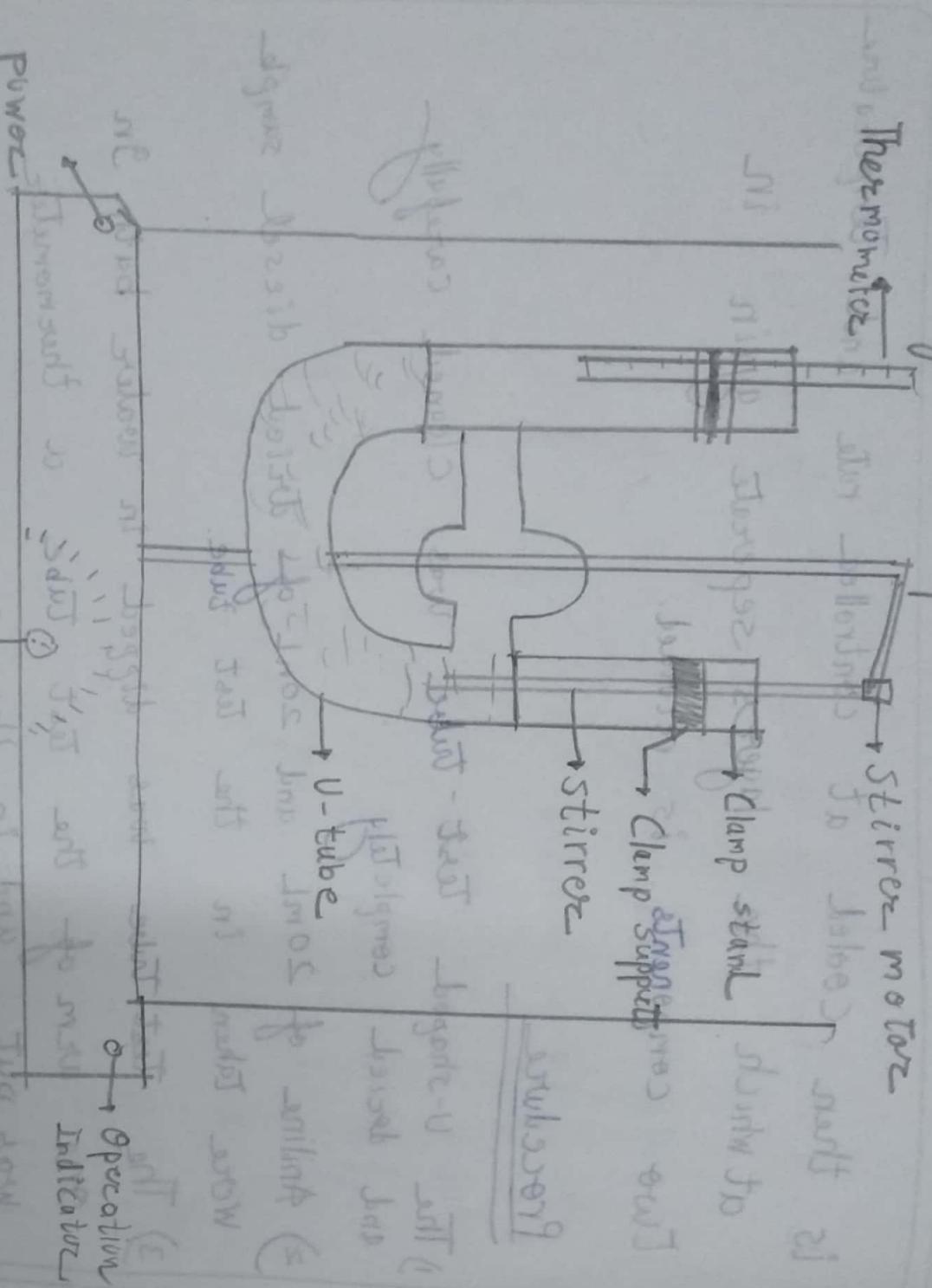
SHEET NO. 32

is then cooled at controlled rate and Temperature at which the layers separate again in two components is recorded.

## Procedure

- 1) The U-shaped test-tube was cleaned carefully and dried completely.
- 2) Aniline of 20mL and 20mL of dried diesel sample were taken in the test tube.
- 3) The test tube was dipped in water bath. In one arm of the test tube a thermometer was put and in the other a mechanical stirrer.
- 4) The test tube was heated consistently at a rate of  $1^{\circ}-3^{\circ}\text{C}/\text{min}$  until the two components are completely miscible. The equilibrium temperature was recorded.
- 5) During heating, the mixture was stirred at regular intervals.
- 6) Then, the test tube was cooled down at uniform rate.

Fig. Apparatus setup for determination  
of melting point



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SHEET NO 33

The Temperature at which Turbidity gain starts to appears is recorded.

The steps (i)-(vi) were performed twice with two different samples.

## Observations

S.No	Temperature (Aniline point) during cooling At complete invisibility of aniline point	Aniline point
(1)	71°C	70°C
(2)	73°C	71°C

## Calculations

$$\text{Average aniline point} = \frac{70^\circ\text{C} + 71^\circ\text{C}}{2} = 70.5^\circ$$

Result-

Aniline point of given sample is  $70.5^\circ$

Discussions :-

1) Aniline point is called "aniline point temperature" which is the lowest temperature ( $^{\circ}\text{F}$  or  $^{\circ}\text{C}$ ) at which equal volumes of aniline ( $\text{C}_6\text{H}_5\text{NH}_2$ ) and the oil form a single phase. The aniline point (AP) correlates roughly with the amount and type of aromatic hydrocarbons in an oil sample.

2) Aniline being an aromatic compound rarely mixes with aromatic so aniline is used for the experiment.

3) low aniline point indicates low diesel index  
 because of high percentage of aromatics.  
 A high aniline point indicates that the fuel is highly paraffinic and hence has a high diesel index & good ignition quality

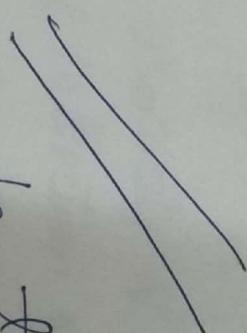
4) The aniline point is a reasonable proxy for aromaticity of oils consisting mostly of saturates

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or unsaturated compounds (mostly aromatic) high aniline point temperature will be high in paraffins and low in aromatics and naphthalenes. Aniline point temperature has also been used to a lower extent in correlative methods for calculating the properties of petroleum fractions such as the heat of combustion, hydrogen content, diesel index and smoke point.



Why aniline is chosen for the aniline miscibility test?

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**Experiment 5(b) - Determination of  
kinematic viscosity of given oil in Redwood viscometer**

SHEET NO. 36

**Objective :-** To determine kinematic viscosity of a liquid fuel sample by Redwood viscometer -

**Theory:** Viscosity of a fluid is measure of its resistance to flow. The ratio of coefficient of viscosity to the density of the fluid is known as kinematic viscosity. The apparatus used is known as redwood viscometer. The kinematic viscosity of oil can be calculated from the formula

$$V = A T - \frac{B}{T}$$

where  $V$  = kinematic viscosity of oil (centistokes)  
 $T$  = time of collection of some sample (seconds)  
 $A, B$  = independent constant

**Apparatus :-**

1. Reduced viscometer
2. 50 ml conical flask
3. Thermometer
4. Stopwatch.

units ?

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SHEET NO 37

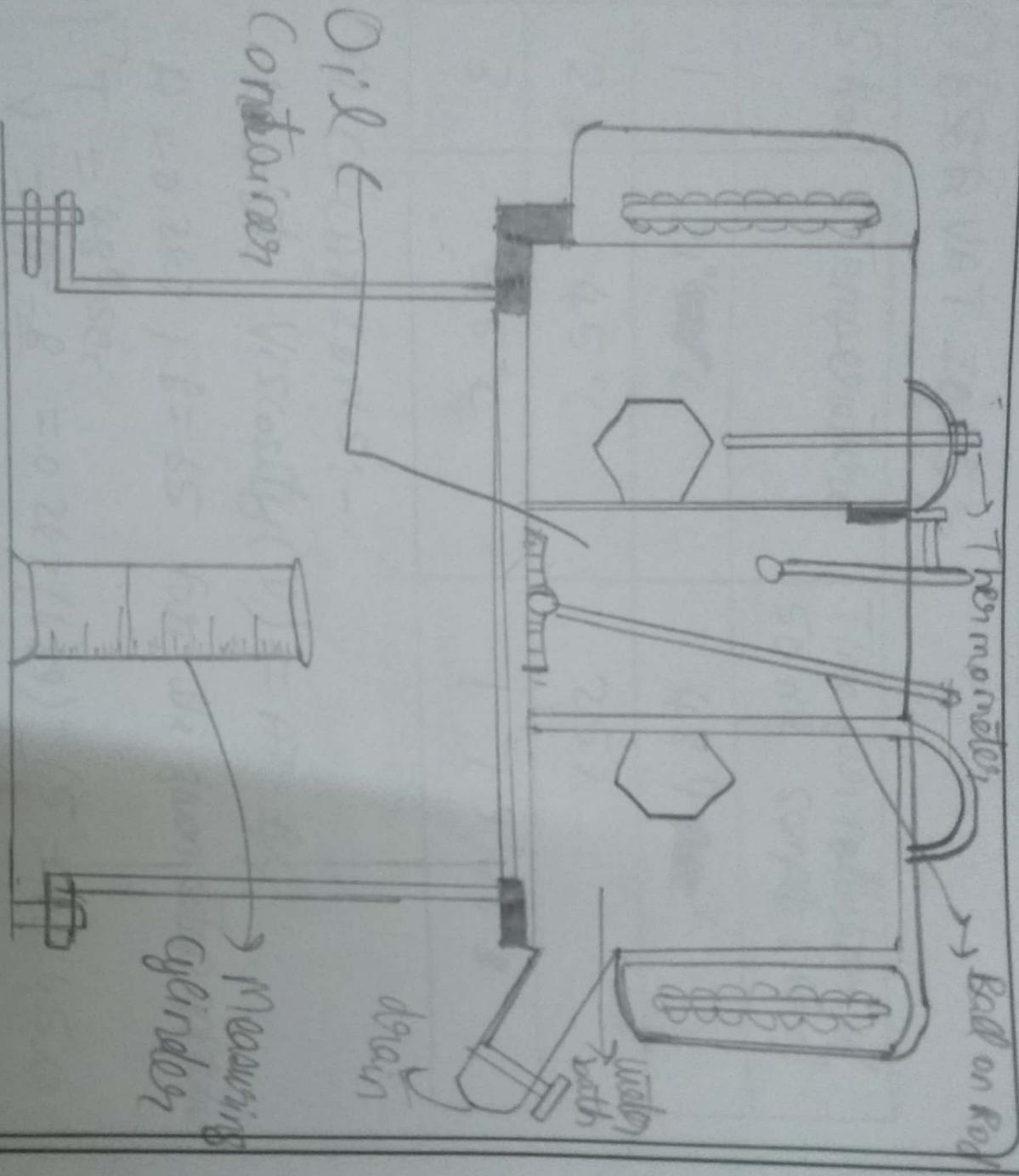


Fig. Reduced Viscosimeter

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SHEET NO. 38

## OBSERVATIONS:-

S.No.	Temperature	Time for collection of 50 ml sample (sec)
1.	31°C	48.9
2.	45°C	25.7
3.	55°C	17.9.26.8

## CALCULATIONS:-

$$\text{Kinematic Viscosity} (\nu) = AT - \frac{B}{T}$$

$A = 0.247$ ,  $B = 65$  for the given setup operating

$$1) T = 489 \text{ sec}$$

$$\nu = AT - \frac{B}{T} = 0.247(489) - \frac{65}{489} = 120.65 \text{ cSt}$$

$$2) T = 257 \text{ sec}, \nu = AT - \frac{B}{T} = 63.22 \text{ cSt}$$

$$3) T = 179.26 \text{ sec}, \nu = AT - \frac{B}{T} = 42.91 \text{ cSt}$$

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SHEET NO. 39

## RESULT :-

S. No. -

	Temp ( $^{\circ}\text{C}$ )	$\gamma (\text{cS})$
1	$31^{\circ}\text{C}$	120.65
2	$45^{\circ}\text{C}$	63.22
3	$55^{\circ}\text{C}$	43.91

## DISCUSSION :-

- There is no change in instrumental constants due to change of test, so that instruments may be calibrated at any convenient temperature, varying the viscosity either by change of temperature or by blending.

- The main disadvantage of the capillary tube viscometer is the errors that variation in the head loss and other parameters, the Hagen Poiseuile formula can still be applied by designing a (efflux type viscometer) that works on the principle of vertical gravity flow of a viscous liquid through a capillary tube.

A reduced viscometer is another offham type viscometer tube. A reduced viscometer is another efflux type viscometer that works on same principle of Saybolt. Here the stopper is replaced with an orifice and reduced seconds is defined for collection of 50 ml of liquid to flow out of surface. Similar expression can be written for reduced viscometer ( $\tau = AT - \frac{B}{T}$ )~~say~~

The error due to change in volume of the oil after it leaves the outlet tube is not major error in viscometry and is negligible at all usual temperature.

Not clear

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Experiment 6 :- Proximate Analysis of Coal.

SHEET NO.41

Aim :- To estimate the contents of a coal sample approximately that means to estimate moisture content, volatile matter and ash content of the coal sample and therefore calculating the fixed carbon content.

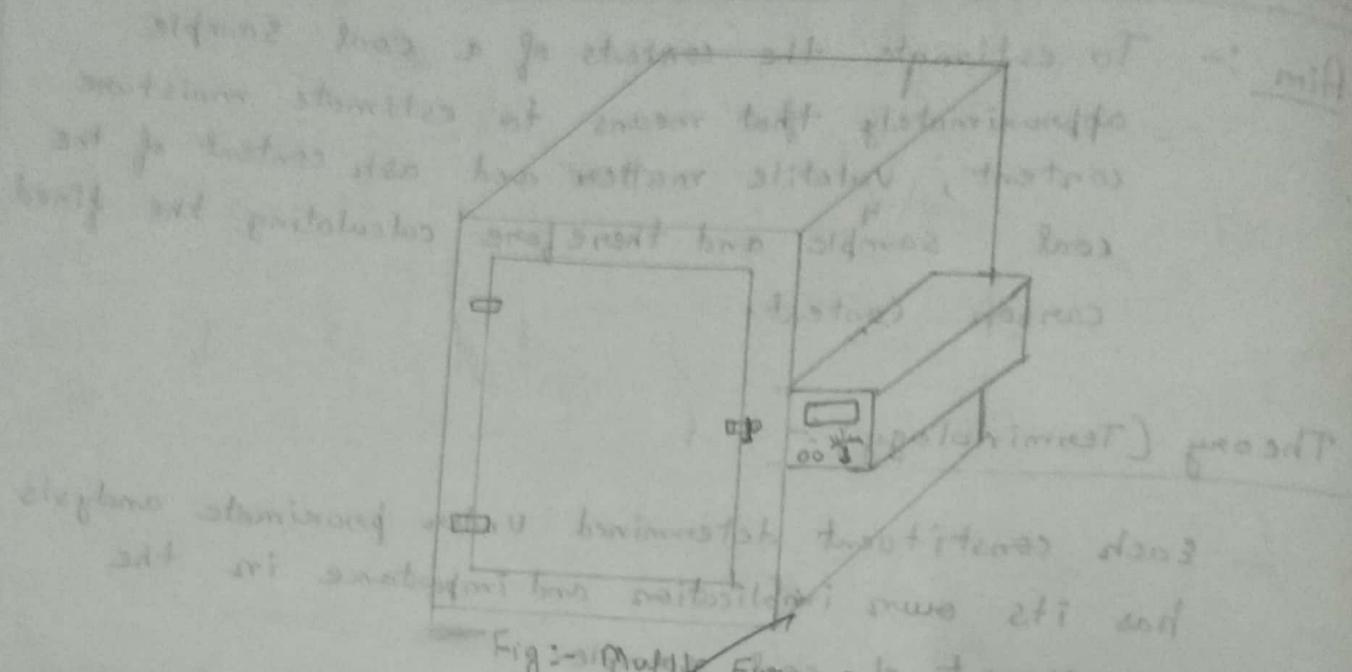
Theory (Terminology) :-

Each constituent determined under proximate analysis has its own implication and importance in the assessment of coal sample.

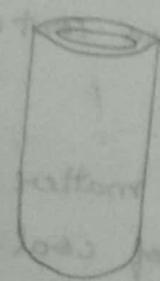
% MOISTURE  $\rightarrow$  Moisture increases the transport cost. Excessive surface moisture may

cause difficulties in handling the coal. Moisture reduces the calorific value. Hence, higher moisture content is undesirable.

% VOLATILE MATTER  $\rightarrow$  The volatile matter is not a constituent of coal but it consists of a complex mixture of gaseous gaseous and liquid products resulting from the thermal decomposition of the coal sample. High volatile matter coal gives long flames, high smoke and relatively low heating values. Coal with low volatile content burns with shorter



transformer set muktikooz shankar et muktikooz  
for muktikooz set jaoz suvachchha etea  
muktikooz shankar et muktikooz et jaoz ghatikooz et muktikooz  
et jaoz suvachchha et jaoz et jaoz suvachchha et  
et jaoz suvachchha



tan et muktikooz shankar et muktikooz et  
et jaoz suvachchha et jaoz et jaoz suvachchha  
hao muktikooz Fig 2-1 Crucible ja shankar et muktikooz  
muktikooz suvachchha et jaoz et muktikooz et jaoz suvachchha  
et jaoz suvachchha et jaoz et jaoz suvachchha  
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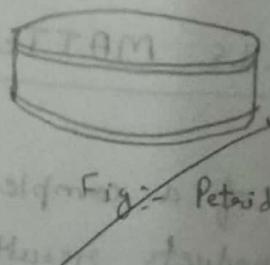


Fig 2-2 Petri dish

flame.

% ASH :→ The ash which intimately interpersed within mass of the coal is called ash. The nature of ash and its amount in coal reduces the heating value of coal. Ash usually consists of a silica-alumina, iron oxide, lime and magnesia.

% FIXED CARBON → The fixed carbon content amount higher in the high grade coals and is also responsible for the calorific value.

APPARATUS :- Electric Oven, muffle furnace, crucible, desicator.

PROCEDURE :-

i) Moisture → 2 gm of coal sample (air dried) in a crucible in an electric oven maintained at  $100-110^{\circ}\text{C}$  is heated for 1 hr. Then, the crucible is cooled down in the desicator and weighed again. The loss in mass gives the moisture content (present on surface).

2) Volatile Content → 1 gm of dried coal sample is placed in the crucible, it is then covered with a lid and placed in an muffle furnace maintained at  $925^{\circ}\text{C}$  for 7 minutes. Next, the crucible is rapidly cooled. Further, loss in mass this time gives the volatile content within the coal.

3) ASH → The residual coal in crucible from step 2 is heated without ~~heat~~ lid in a muffle furnace at  $750^{\circ}\text{C}$  for  $\frac{1}{2}$  hr. The crucible is cooled down in desiccated desicator and weighed again. The mass of residue gives ash content.

4) Fixed Carbon → The sum total of the percentage of volatile matter, moisture and subtracted from 100, gives the percentage of fixed carbon.

### OBSERVATIONS :-

Moisture Content	
wt. of empty crucible ( $w_1$ ) g	47.871 g
wt. of crucible + coal sample ( $w_2$ ) g	48.871 g
wt. of crucible + sample after heating ( $w_3$ ) g	49.857 g

Volatile Content

wt. of empty crucible ( $w_1'$ ) g	13.863 g
wt. of crucible + coal sample ( $w_2'$ ) g	14.863 g
wt. of crucible + sample after heating ( $w_3'$ ) g	14.725 g

Ash Content

wt. of empty crucible ( $w_1''$ ) g	15.043 g
wt. of crucible + coal sample ( $w_2''$ ) g	16.043 g
wt. of crucible + sample after heating ( $w_3''$ ) g	15.311 g

CALCULATIONS :-

$$1) \% \text{ Moisture} = \frac{w_2 - w_3}{w_2 - w_1} \times 100 = \frac{49.871 - 49.857}{2} \times 100 \\ = 0.7\%$$

$$2) \% \text{ volatile content} = \left[ \frac{w_2' - w_3'}{w_2' - w_1'} - \text{moisture content} \right] \times 100 \\ = \left[ \frac{14.863 - 14.725}{1} - \frac{0.7}{100} \right] \times 100 \\ = 13.7\%$$

$$3) \% \text{ Ash Content} = \frac{w_2'' - w_3''}{w_2'' - w_1''} \times 100 = \frac{16.048 - 15.316}{1} \times 100 \\ = 26.7\%$$

$$4) \text{ Fixed Carbon} = 100 - (0.7 + 26.7 + 13.7) \\ = 58.9\%$$

RESULT :-

$$\text{Percentage of fixed carbon} = 58.9\%$$

DISCUSSION :-

Coal analysis techniques are specific methods designed to measure different properties of coal. The three traditional techniques are proximate analysis, ultimate analysis and calorific or heating value.

Proximate analysis is used for measurement of moisture, ash, volatile matter and fixed carbon. This technique has been widely used for over 160 years.

Moisture content is determined by mass loss that a coal sample undergoes after it has been heated to high temperature.

Under  $N_2$  atmosphere. The moisture content measured ~~water~~ represents water that may be physically or chemically bound in the coal with the exception of mineral hydrates that decomposes above  $110^\circ C$ . Anthracite has least quantity of moisture whereas lignite has the highest.

~~The volatile matter content of coal corresponds to the volatile products evolved between  $110 - 925^\circ C$  under  $N_2$  as a result of thermal decomposition. Volatile matter content varies with the rank of the coal.~~

Fixed Carbon is the solid combustible material that remains after the loss of moisture and volatile matter minus the ash that remains after combustion is complete. Fixed carbon content decreases with the decrease in quality of coal.

Ash is residue that remains after the coal sample has undergone loss of moisture and volatile matter and the fixed carbon has undergone combustion at  $925^\circ C$  in air.

~~On the basis of above factor coals are classified in five types :- A, B, C, D, E.~~

~~Anthracite is the best quality of coal known till date followed by bituminous, lignite and peat.~~

20/10/22  
Bachir

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20/10/22

Experiment-7(a)

Determination of cloud point and pour point of heavy oil.

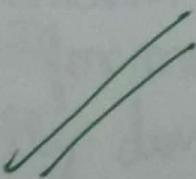
SHEET NO.

## I) Aim of experiment:-

To determine the cloud point and pour point of a heavy oil. This experiment is performed to ensure safety of cross continent crude oil pipelines.

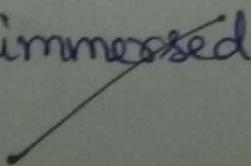
## II) Apparatus required:-

- i) Jars
- ii) Cork
- iii) Analog thermometer
- iv) Methanol bath cooling system.



## III) Procedure:-

- i) Oil was poured in the test jar upto the level mark.
- ii) The test jar was then closed with the cork carrying the thermometer.
- iii) The thermometer bulb was immersed in the oil sample.

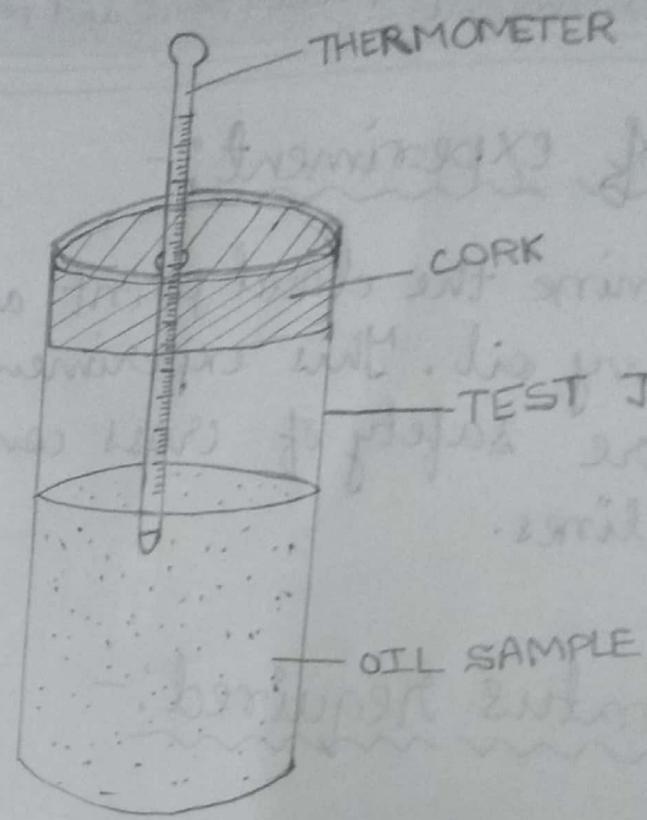


- iv) The jar was then placed in the cooling bath and the temperature of the bath was maintained within the range of the pour point of the oil.
- v) After the oil was cooled enough, the jar was carefully removed from the bath and was tilted in such a way as to observe whether there was movement of oil in the test jar.
- vi) The complete operation of removal and replacement was not taken for more than a minute. This was carefully done several times.

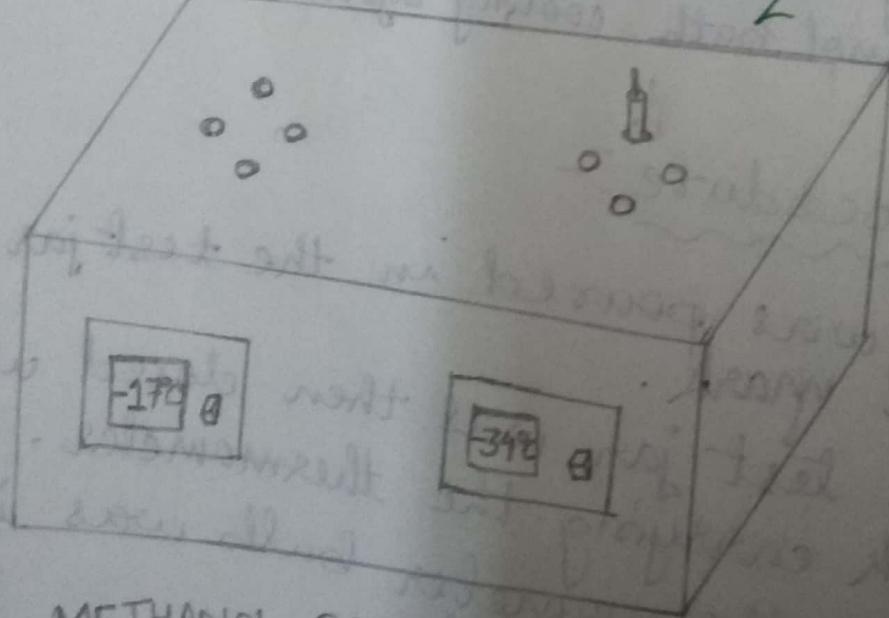
## IV) Observation Table :-

SN No.	Cloud point ( $^{\circ}\text{C}$ )	Pour Point ( $^{\circ}\text{C}$ )
1	$1^{\circ}\text{C}$	$-24^{\circ}\text{C} + 3^{\circ}\text{C} = -21^{\circ}\text{C}$
2	$4^{\circ}\text{C}$	$-15^{\circ}\text{C} + 3^{\circ}\text{C} = -12^{\circ}\text{C}$

why  $+3^{\circ}$  ?



OIL SAMPLE IN TEST JAR



METHANOL BATH COOLER

## v) Discussions:-

### i) Cloud point:-

Cloud point refers to the temperature below which a solution undergoes a liquid-solid phase transition to form a stable sd or suspension that settles as a precipitate as a result of which a cloudy appearance is observed.

### ii) Pour point:-

Pour point is a measure of the tendency of a fuel to become more viscous under prescribed temperature conditions and resist flowing when cold.

## vi) Importance:-

The cloud point is important for determining storage stabilities while pour point is the lowest temperature at which oil is capable of flowing under gravity. It is an important low temperature characteristic.

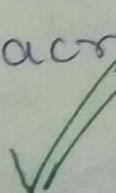
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QUESTION 5

of high boiling fractions. When the surrounding temperature is lower than the pour point of a petroleum product, it can neither be stored nor transferred through pipelines.

If there are regions where the temperature is very low and the oil needs to be transferred through pipelines in these regions, the oil may freeze. One way is to heat them but it is a very expensive task. Alternatively we can add some substances to increase its pour point to make it easier to transport the oil across the low temperature regions.



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Experiment - T(b)

Determination of caking index of coal.

SHEET NO. 51

## I) Aims of experiment:-

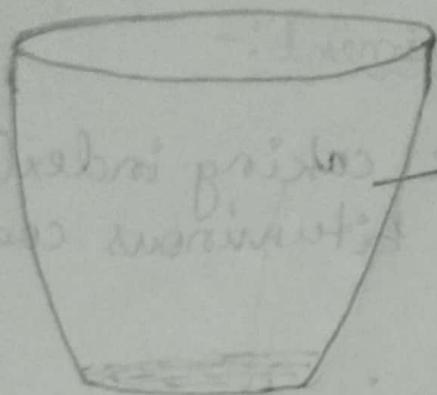
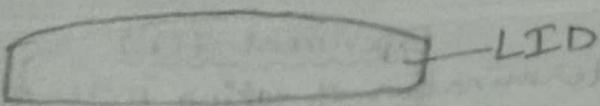
To determine caking index (bonding property) of a bituminous coal.

## II) Apparatus required:-

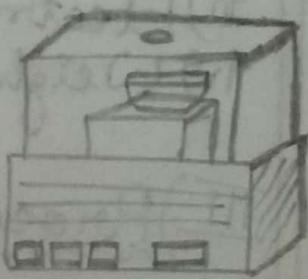
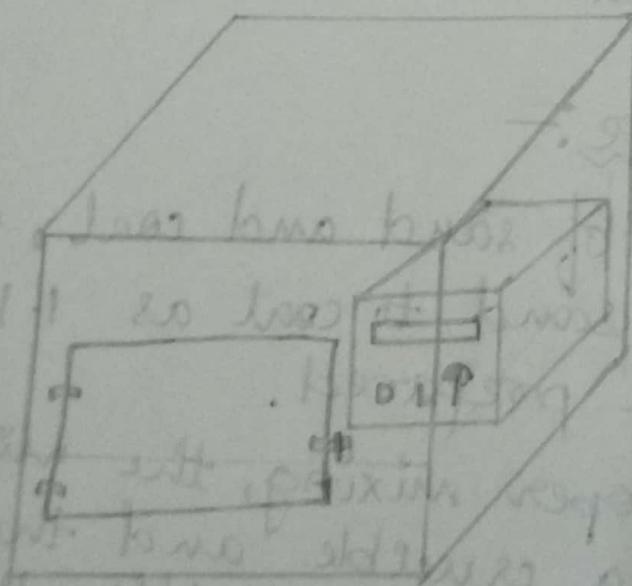
- i) Alumina crucible
- ii) Lids
- iii) Electric muffle furnace.
- iv) Weight box.

## III) Procedure:-

- i) 4 mixtures of sand and coal, each having ratio of sand to coal as 1:1, 2:1, 3:1, 4:1, 7:1 were prepared.
- ii) After proper mixing, the mixtures were taken in a crucible and the crucibles were kept in a muffle furnace at a temperature of  $925^{\circ}\text{C}$  for 7 minutes.
- iii) The crucibles are then taken out, allowed to cool and the obtained mass is then



CRUCIBLE



WEIGHT  
BOX

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SHEET NO. 52

separated from the crucible.

- iv) Over each cake, a 500gram weight is placed which causes to break the loose materials.
- v) These are collected and weighed.
- vi) Then the percent of loose grains are plotted against corresponding sand to coal ratio and from this graph, coking index was determined.

## IV) Observation Table:-

The composition of the components within the crucible are as follows:-

- i) 12.5g of sand and 12.5g of coal in a ratio of 1:1.
- ii) 16.67g of sand and 8.34g of coal in a ratio of 2:1.
- iii) 18.75g of sand and 6.25g of coal in a ratio of 3:1.
- iv) 20g of sand and 5g of coal in a ratio of 4:1.

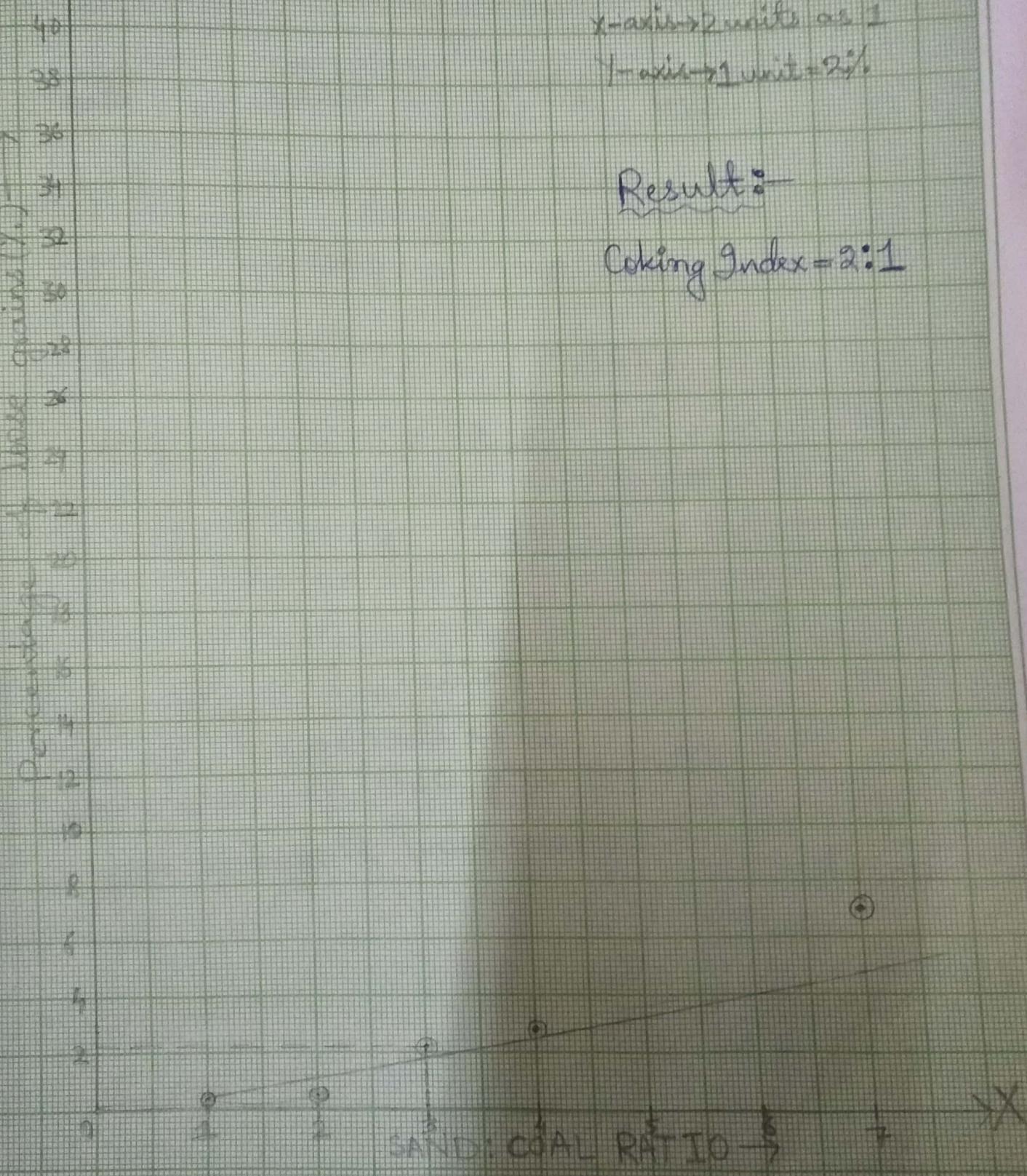
SCALE :-

X-axis  $\rightarrow$  2 units as 1

Y-axis  $\rightarrow$  1 unit = 2%

Result :-

Coking Index = 2:1



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SHEET NO. 53

Weight ratio	Weight obtained	% loose grains = $\frac{\text{weight}}{\text{total weight}} \times 100\%$
1:1	0.03 g	0.12%
2:1	0.04 g	0.16%
3:1	0.542 g	2.168%
4:1	0.775 g	3.10%
7:1	1.875 g	7.5%

//

I) Sample calculations:-

For sand to coal ratio as 1:1.

i) Weight of loose grains = 0.03 g

$$\text{percentage of loose grains} = \frac{\text{weight obtained}}{\text{total weight}} \times 100$$

$$= \frac{0.03 \text{ g}}{25 \text{ g}} \times 100 = 0.12\%$$

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ii) For sand to coal ratio as 2:1.

$$\text{weight obtained} = 0.04 \text{ g}$$

$$\text{Total weight} = 25 \text{ g}$$

$$\text{percentage of loose grains} = \frac{0.04}{25} \times 100 \\ = 0.16\%$$

iii) For sand to coal ratio as 3:1.

$$\text{weight obtained} = 0.542 \text{ g}$$

$$\text{Total weight} = 25 \text{ g}$$

$$\text{percentage of loose grains} = \frac{0.542}{25} \times 100 \\ = 2.168\% \\ \approx 2.17\%$$

iv) For sand to coal ratio as 4:1.

$$\text{weight obtained} = \cancel{6.014} \text{ g } 0.775 \text{ g}$$

$$\text{Total weight} = 25 \text{ g}$$

$$\text{percentage of loose grains} = \frac{0.775}{25} \times 100 \\ = 3.1\%$$

V) For sand to coal ratio as 7:1.

$$\text{weight obtained} = \cancel{8.4g} \quad 1.875g$$

$$\text{Total weight} = 25g$$

$$\text{percentage weight} = \frac{\cancel{1.875g}}{\cancel{25g}} \times 100$$

So what is the caking index for this sample?  $= 7.5\%$

VI) Discussions:-

The caking index of a coal is a measure of binding strength between coal particles and/or between coal particles and inert particles after the mixture of coal and inert particles is heated at  $925^{\circ}\text{C}$ . Bituminous coal must meet a set of criteria for use as a caking coal. This can be determined by particular coal assay techniques. These techniques determine

the moisture content, ash content, sulphur content, volatile content, tar and plasticity of a coal.

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Coking coal is different from thermal coal but it differs not by coal forming process. Coking coal has different macerals from thermal coal. The different macerals are related to source of materials that compose the coal. However, the coke is of widely varying strength and ash content and generally considered unsellable except in some cases as a thermal product.

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The caking index has been widely popularized and applied in coal geological exploration, production and utilization departments in China. This index was established after improving the Roga Index, an international method used for

determining the caking power of bituminous coal. It has become one of the main indices to classify bituminous coal in the new classification of Chinese coals.

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Exp. 8: Determination of Gross calorific value of a given sample of coal by Bomb Calorimeter

SHEET NO. 58

## Aim of the Experiment:

To determine the <sup>gross</sup> calorific value of a given sample of coal by Bomb Calorimeter.

## Theory:-

Calorific value is an important characteristic of a fuel. For it expresses the amount of heat generated by complete combustion of unit weight of fuel. The determination requires special apparatus and a standardized procedure in which an accurate weight of fuel (about 1 gm) is burned in an atmosphere of compressed oxygen in a calorimetric bomb immersed in water, the temperature rise of the water being measured with extreme accuracy.

The laboratory determination of calorific value using bomb calorimeter gives a measure of the Gross calorific value at constant volume. When coal is burnt for industrial purposes no element of pressure within a container (such as the bomb) is involved; the operative calorific value (C.V.) is the C.V. at const. pressure. Difference betw the two is very small.

Gross Calorific value is determined by following eqn:-

$$C.V. = \frac{1}{m} \left\{ (W+E)C_p T - m_1 (C.V.)_{\text{string}} - m_2 (C.V.)_{\text{wire}} \right\}$$

where,

W = weight of water

E = water equivalent of calorimeter

C<sub>p</sub> = sp. heat of water

T = Rise of temp.

m<sub>1</sub> = mass of string

m<sub>2</sub> = mass of wire

m = mass of sample

for calorimetry may be determined  
with direct fuel to air ratio = 1.0

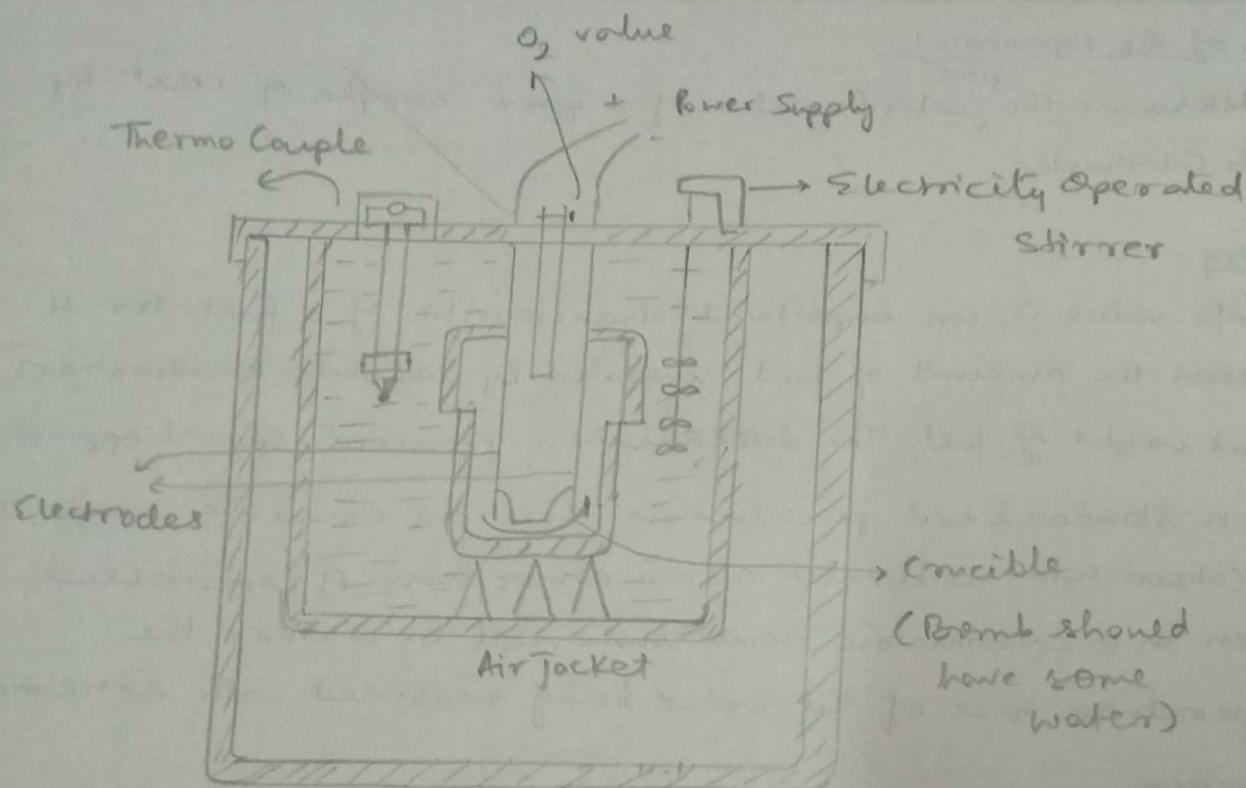


Fig: Schematic Diagram of  
Bomb Calorimeter

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SHEET NO. 59

## Apparatus :-

Bomb Calorimeter  
Backman thermometer  
Stop watch  
Weight box  
Measuring cylinder  
Firing wire  
Cotton thread

## Procedure :-

Sufficient finely ground (60 mesh 1 mm sieve) air-dried coal is compressed into a cylindrical pellet and weighed in to the concible of the calorimeter. A piece of fine platinum <sup>nickel chrome</sup> wire is tightly stretched across the pale pieces of the bomb and one end of a piece of sewing cotton is tied around the wire; the concible is placed in position and the loose end of the cotton is arranged so as to be in contact with the coal.

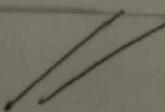
About 15 ml of distilled water is introduced in to the bomb & the bomb is connected with an oxygen cylinder. Oxygen is slowly supplied; the pressure is being allowed to rise to 25 atm. After being disconnected from the filling apparatus the bomb is introduced in to the calorimeter vessel with sufficient water inside to submerge the cover of the bomb. The stirrer is then started.

After that, the ends of the firing wires are connected to ignite the charge and readings are taken at 30 seconds interval. Then temp is first found to rise then fall with time. Readings are continued till the rate of fall is uniform.

Observations :-

S.No.	Quantity	Magnitude
1.	Weight of water inside the calorimeter assembly	1800 ml
2.	Water inside Bomb	15 ml
3.	Weight of string	
4.	Weight of wire	$0.039 \text{ gm} = m_1$
5.	Oxygen gas pressure	$0.017 \text{ gm} = m_2$
6.	Weight of coal pellet	$25 \text{ kg/cm}^3$
7.	Calorific Value of wire	$1.05 \text{ gm} = m$
8.	Calorific value of string	$335 \text{ cal/g}$
9.	Water Equivalent of bomb Calorimeter	$4000 \text{ cal/g}$ $450 \text{ gm}$

Table - I



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S.No	Time (sec)	Temp (°C)
1	0	0
2.	30	0.85
3.	60	1.55
4.	90	1.94
5.	120	2.18
6.	150	2.30
7.	180	2.39
8.	210	2.44
9.	240	2.48
10.	270	2.50
11.	300	2.52
12.	330	2.54
13	360	2.54
14	390	2.55
15	420	2.55
16	450	2.56
17	480	2.56
18	510	2.55
19	540	2.55
20	570	2.54
21	600	2.54

Graph: Temp( $^{\circ}\text{C}$ ) v/s Time(s)

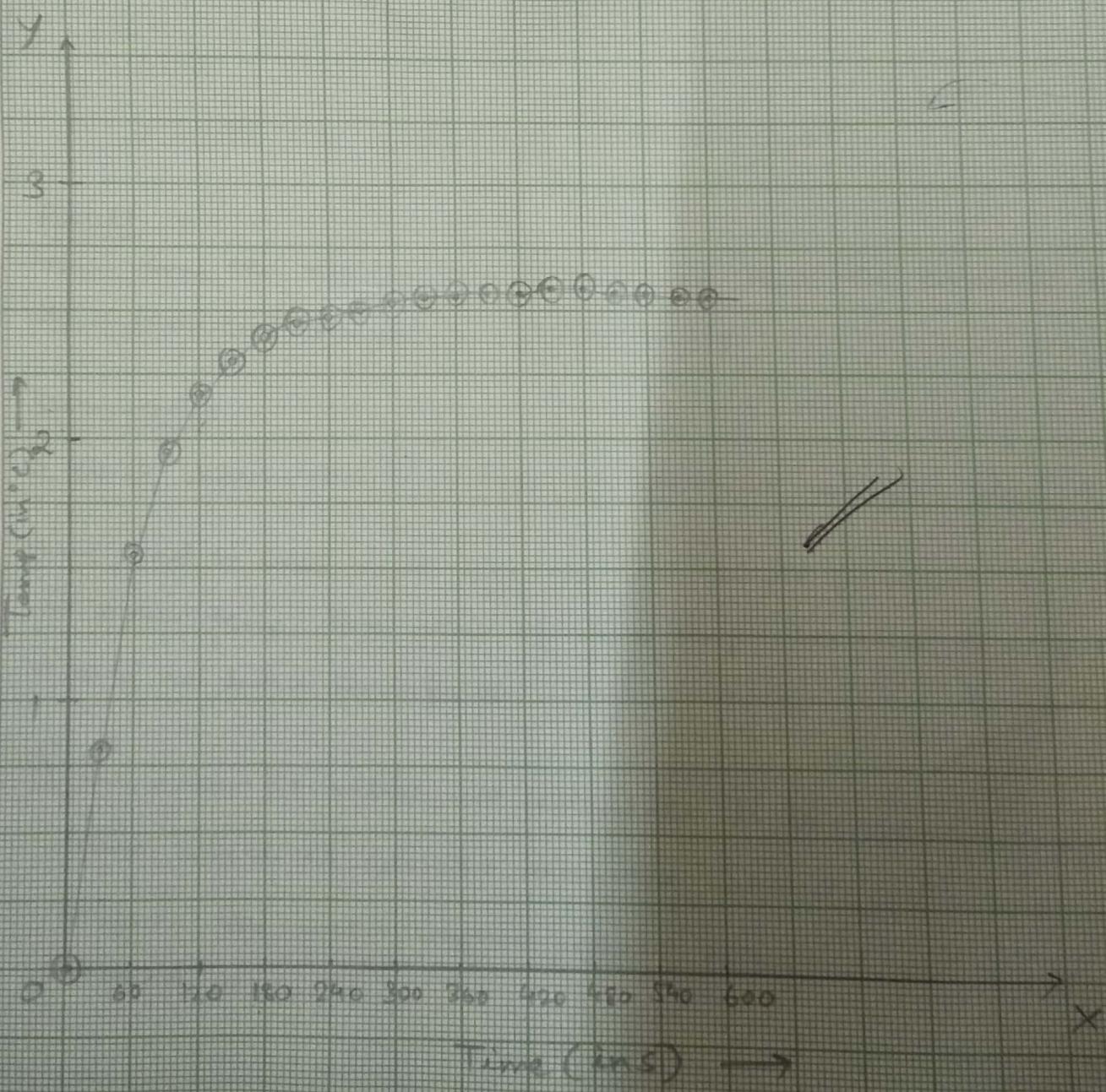
Slope

x axis

1 unit = 60 s

y axis

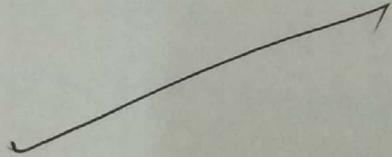
1 unit =  $0.25^{\circ}\text{C}$



Calculations :-

$$T = 2.56^\circ\text{C}$$

$$\begin{aligned} \text{C.V.} &= \frac{1}{m} \left\{ (W+E) C_p \times T - m_1 (\text{C.V.})_{\text{string}} - m_2 (\text{C.V.})_{\text{wire}} \right\} \\ &= \frac{1}{1.05} \left\{ (1800 + 450) \times 1 \times 2.56 - 0.039 \times 4000 - 0.017 \times 335 \right\} \\ &= \frac{1}{1.05} \left\{ 5760 - 156 - 5.695 \right\}, \\ &= 5331.719 \text{ cal/g.} \end{aligned}$$



Result :-

Thus, from the experiment we obtained that the calorific value of a given coal sample is 5.3317 Kcal/g

Discussion :-

- 1) Bomb calorimeter is a type of constant volume-calorimeter.
- 2) We need to make sure that the coal sample provided isn't at higher temp. than that of room temp. because it is possible, it may not burn henceforth.
- 3) The calorific value we calculate is the gross calorific value which is greater than the net calorific value as because the water vapour present release heat when allowed to condense.

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- 4) Oxygen is supplied at high pressure so we need to ensure that the pipe connecting the oxygen cylinder is not damaged, because it may lead to bursting of the pipe.
- 5) 10-15ml of water present inside is to absorb the by-product gases such as  $\text{SO}_2$ , etc.
- 6) According to the Indian grading system based on G.C.V., the grade of the coal sample we used is G-7 (5200-5500  $\text{Kcal/kg}$ )
- 7) In Gross calorific value exp., the water produced during combustion is allowed to condense whereas in net calorific value exp. the water remains in the gaseous state.  
Discussion is mostly ~~opted~~ similar ~~for~~ with other groups.