GOVERNMENT POLYTECHNIC

BHATAPARA

LAB MANUAL FOR

APPLIED CHEMISTRY

SUBJECT CODE – 2000291(011)

SESSION: ____________

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GUIDED BY

SUBMITTED BY

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S.NO	EXPERIMENT	DATE OF EXPERIMENT	DATE OF SUBMISSION	REMARK
1	**To measure the pH of different solutions by pH paper/pH meter.**			
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3	**To estimate proximate analysis of coal sample.**			
4	**To determine the percentage of iron in iron salt by redox titration.**			
5	**To determine the percentage of copper in given sample of brass iodometrically.**			
6	**To determine the kinetic viscosity of the given sample of oil of various temperature and to study corresponding variation with respect to temperature**			
7	**To determine the flash and fire point of a given bituminous material.**			

MAPPING OF POs, PSOs and CO

Course Outcomes (COs)	Programme Outcomes (POs)										Programme Specific Outcomes (PSOs)	
	PO-1 Basic knowledge	PO-2 Discipline knowledge	PO-3 Experiments and practice	PO-4 Engineering Tools	PO-5 The engineer and society	PO-6 Environment and sustainability	PO-7 Ethics	PO-8 Individual and team work	PO-9 Communication	PO-10 Life-long learning	PSO-1	PSO-2
CO-1 Solve various engineering problems applying the basic knowledge of atomic structure and chemical bonding.	3	1	1	-	-	-	1	2	2	1	-	1
CO-2 Use relevant water treatment method to solve industrial problems.	3	1	3	3	2	2	1	3	1	2	1	1
CO-3 Solve the engineering problems using concept of Electrochemistry	3	1	2	2	1	2	-	3	2	2	-	1
CO-4 Solve the engineering problems by applying the knowledge of metallurgical process and Metals Alloys	3	1	3	3	-	2	-	3	1	2	1	1
CO-5 Use relevant fuel and lubricants for industrial applications.	3	1	3	2	1	1	-	2	1	2	1	1

Legend: 1 – Low, 2 – Medium, 3 – High

Lab Safety Dos and Don'ts for Students

Conduct

- Do not engage in practical jokes or boisterous conduct in the laboratory.
- Never run in the laboratory.
- The use of personal audio or video equipment is prohibited in the laboratory.
- The performance of unauthorized experiments is strictly forbidden.
- Do not sit on laboratory benches.

General Work Procedure

- Know emergency procedures.
- Never work in the laboratory without the supervision of an instructor.
- Always perform the experiments or work precisely as directed by your instructor.
- Immediately report any spills, accidents, or injuries to your instructor.
- Never leave experiments while in progress.
- Never attempt to catch a falling object.
- Be careful when handling hot glassware and apparatus in the laboratory. Hot glassware looks just like cold glassware.
- Never point the open end of a test tube containing a substance at yourself or others.
- Never fill a pipette using mouth suction. Always use a pipetting device.
- Make sure no flammable solvents are in the surrounding area when lighting a flame.
- Do not leave lit Bunsen burners unattended.
- Turn off all heating apparatus, gas valves, and water faucets when not in use.
- Do not remove any equipment or chemicals from the laboratory.
- Store coats, bags, and other personal items in designated areas.
- Notify your instructor of any sensitivity that you may have to particular chemicals.
- Keep the floor clear of all objects (e.g., ice, small objects, and spilled liquids).

EXPERIMENT NO. 1

Aim:-

To measure the pH of different solutions by pH paper/pH meter.

Material Required:-

Digital pH meter, beaker, different solutions, buffer solutions, pH paper etc.

Principle:

The pH scale measures how acidic or basic a substance is. The pH scale ranges from 0 to 14. A pH of 7 is neutral. A pH less than 7 is acidic. A pH greater than 7 is basic. Water molecules exist in equilibrium with hydrogen ions and hydroxide ions.

$$H_2O \text{ <--> } H^+ + OH^-$$

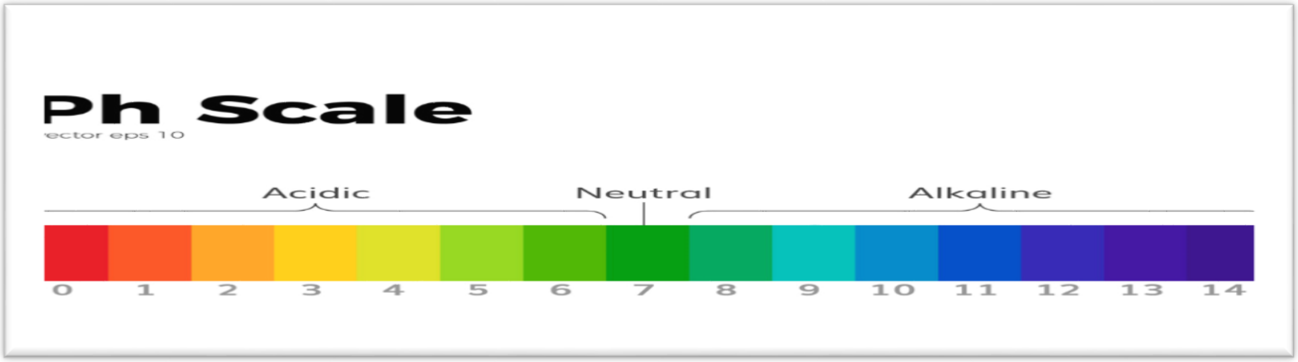
The water equilibrium constant is written as:

$$Kw = [H^+]\,[OH^-]$$

Experimentally, it has been found that the concentration of:

$$H^+ = OH^- = 10^{-7}$$

Therefore: $Kw = [10^{-7}][\,10^{-7}] = [10^{-14}]$



The values for Kw, H^+, OH^- concentration all indicate that the equilibrium favors the reactant (water molecules). In other words, only very small amounts of H^+ and OH^- ions are present.

The concentrations of hydrogen ions and indirectly hydroxide ions are given by a pH number. **pH is defined as the negative logarithm of the hydrogen ion concentration.** The equation is:

$$\mathbf{pH = - \log [H^+]}$$

Method:-

1. Turn the pH meter on and allow adequate time to warm up (check the operating manual).
2. Select two pH buffers that bracket the expected sample pH. The first buffer should be pH 7.00 (zero point adjustment) and the second buffer should be near the expected sample pH (pH 4 or pH 9.21). If you will measure both acidic and basic samples, and only perform one pH calibration, you should use a multi-point calibration.
3. Before starting calibration, be sure the sensor and the buffer solution are at the same temperature. If not, allow time for temperature equilibration.
4. Pour the necessary amount of buffer solutions into individual glass beakers. Buffer solutions will remain stable in a glass beaker for a maximum of 2 hours. Note: To minimize the risk of contamination, calibrations should never be carried out in the storage container.

   Close the buffer containers promptly to avoid carbon dioxide absorption. Do not pour used buffer back into the storage container. Discard it.
5. Place the electrode into the first buffer. When the reading is stable, set the pH meter to the pH value of the first buffer at the measured temperature. Most modern pH meters have an "auto-read" function for early detection of a stabilized reading.

   It is not strictly necessary to stir samples for pH measurement. However, if the buffer solutions are stirred, then the sample should also be stirred in the same way.
6. Between buffers, rinse the electrode with distilled water and then with the next buffer.

   If you, for some reason, not want to rinse the pH electrode with the next buffer; Rinse the electrode with distilled water and gently blot it dry with a lint-free tissue. Avoid rubbing or wiping the electrode bulb.
7. Repeat step 3 for the next buffer.
8. When the pH meter calibration is done, rinse the electrode and place into the sample and make your pH measurement.

Observations:-

S.NO.	SOLUTIONS	pH VALUE

Result:-

pH of solution no. 1-

pH of solution no. 2-

pH of solution no. 3-

pH of solution no. 4-

pH of solution no. 5-

Precautions:-

1- The electrode should be calibrated before use.
2- Buffer solution should be stored in cool place.
3- Never touch a sensor electrode or reference cell with your fingers

EXPERIMENT NO. 2

Aim:-

To determine the hardness of given water sample by EDTA method.

Materials Required:-

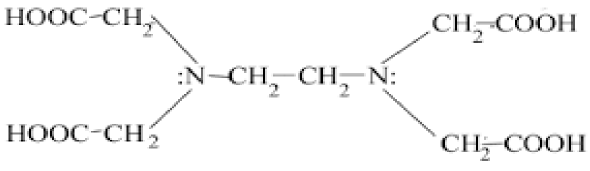
EDTA solution, water sample A &B, buffer solutions (pH 10), erichrome black T (EBT) etc. Burete,pipette, conical flask, glassrod,beaker, dropper etc.

Principle:-

Water hardness is usually noticed because of difficulty in lathering soap and the formation of a scum in the bathtub. Ca2+ and Mg2+ form insoluble salts with soaps causing precipitation of the soap scum. Another effect of hard water is "boiler scale". When hard water comes into contact with dissolved carbonates, a precipitate of insoluble calcium carbonate forms. This "scale" can build up on the inside of water pipes to such a degree that the pipes become almost completely blocked.

The ions involved in water hardness, i.e. Ca2+(aq) and Mg2+(aq), can be determined by titration with a chelating agent, ethylenediaminetetraacetic acid (EDTA), usually in the form of disodium salt (H2Y2-). The titration reaction is:

$Ca^{2+}(aq) + H_2Y^{2-}(aq) \longrightarrow CaY^{2-(}aq) + 2H^+(aq)$



structure of EDTA

Eriochrome Black T is commonly used as indicator for the above titration. At pH 10, $Ca^{2+}(aq)$ ion first complexes with the indicator as $CaIn^+(aq)$ which is wine red. As the stronger ligand EDTA is added, the $CaIn^+(aq)$ complex is replaced by the $CaY^{2-}(aq)$

Complex which is blue. The end point of titration is indicated by a sharp colour change from wine red to blue.

Titration using Eriochrome Black T as indicator determines total hardness due to Ca^{2+}(aq) and Mg^{2+}(aq) ions.

Procedure:-

1-Wash the pipette, burette and conical flask with deionized water. Rinse the burette With the EDTA solution and the pipette with the hard water.

2-Using the funnel, fill the burette with the EDTA solution. Open the tap briefly to fill the part below the tap. Remove the funnel. Adjust the level of the solution to the zero mark. Make sure that the burette is vertical.

3-Use the pipette to transfer 50 cm^3 of the hard water sample to the conical flaskAdd 2-3 cm^3 of the buffer (pH 10) solution (measured out using the graduatedcylinder).

4-Add 0.03 g of the solid indicator to the contents of the flask in the following manner: Add gradually to the flask, swirling after each addition. A deep wine red colour is obtained.

5-Carry out one 'rough' titration to find the approximate end point, followed by a number of accurate titrations until two titres agree to within 0.1 cm^3. At the end point, the colour should be dark blue, with no tinge of wine-red colour.

6-From the data, calculate the total hardness of the water sample.

Observation and Calculations:-

Sample “A” or known hard water-----

S.NO.	VOLUME OF SAMPLE “A”	BURETE READING OF EDTA SOLUTION		VOLUME OF EDTA (in ml)
		INITIAL	FINAL	

$$N_1V_1 = N_2V_2$$

sample "B" or Unknown hard water-----

S.NO.	VOLUME OF SAMPLE "A"	BURETE READING OF EDTA SOLUTION		VOLUME OF EDTA (in ml)
		INITIAL	FINAL	

$$N_2V_2 = N_3V_3$$

Result:-

The total hardness of given unknown water sample "B" is found to be _________ppm.

Precautions:-

1. Reading should be taken carefully.

2. The end point should be checked.

3. Note that there should be no air bubble in burette.

EXPERIMENT NO. 3

Aim:-

To estimate proximate analysis of coal sample.

Material required:-

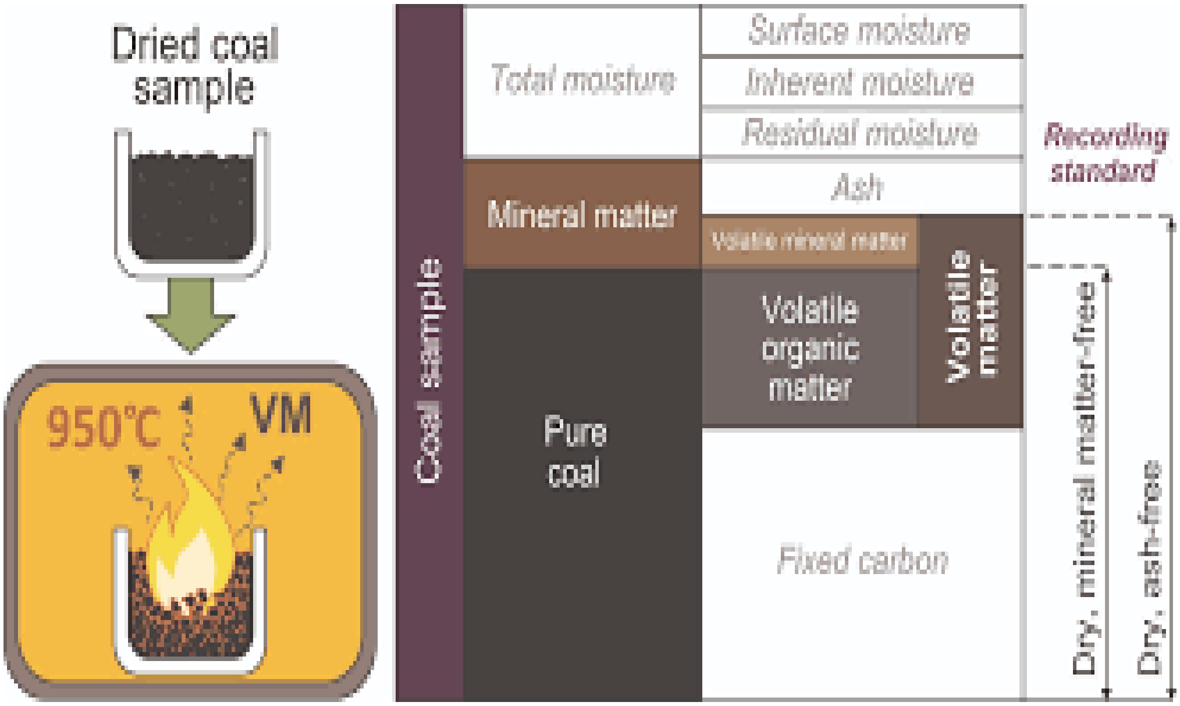
Coal, silica crucible, Bunsen burner,desiccator etc.

Principle:-

Coal is a fossil fuel which occurs in layers in the earth's crust. Coal is an important fuel. The coal varies widely and hence it is necessary to analyse. The quality of a coal is ascertained by two types of analysis.

The proximate analysis which includes the determination of moisture, volatile matter, ash and fixed carbon. This gives quick and valuable information regarding commercial classification and determination of suitability for a particular industrial use.

The ultimate analysis which includes the estimation of ash, carbon, hydrogen, sulfur, nitrogen and oxygen. The ultimate analysis is essential for calculating heat balances in any process for which coal is employed as a fuel.



Procedure for Proximate Analysis

1. Moisture:- Take a weight of empty silica crucible then take about 1 gm of the air dried coal sample in it and again weigh it accurately. Now, heat the crucible in an oven maintained at a temperature of 105°C to 110°C for 1 hour. Cool the crucible in a desiccator and weigh it again. The loss in weight correspond to the moisture.

2. Volatile Matter: - The dried sample of coal left in the crucible is then covered with a lid and place in an electric furnace (Muffle furnace) maintained at 925°C for 7 minutes. Cool the crucible in a desiccator. Weigh the crucible with the contents again. The loss in weight is reported as volatile matter.

3. Ash: - The residual coal in the crucible is then heated without lid in Muffle furnace at 750°C for 1/2 hour. Cool the crucible in desiccator and weight of the residue remaining in the crucible corresponds to the ash content of the coal.

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

Calculation

1. Moisture: - Weight of the empty crucible (W1) = _______ gm

Weight of crucible + Coal sample (W2) = _______ gm

Weight of crucible + Sample after heating (W3) = ______ gm

$$\textbf{\% Moisture} = \frac{\mathbf{(W2 - W3) \times 100}}{\mathbf{(W2 - W1)}}$$

2. Volatile Matter: - Weight of the empty crucible (W1) = _______ gm

Weight of crucible + Coal sample (W2) = _______ gm

Weight of moisture free coal sample (W3) = ______ gm

Weight of crucible + Sample after heating (W4) = ______ gm

$$\textbf{\% volatile matter} = \frac{\mathbf{(W3 - W4) \times 100}}{\mathbf{(W2-W1)}}$$

Result:-

The percentage of moisture in the given coal sample is found to be ………………… %

The percentage of volatile matter in the given coal sample is found to be ………………… %

Precautions:-

1. The temperature should be maintained accurately.
2. For cooling, the coal sample should be place in desicator.

EXPERIMENT NO. 4

Aim:-

To determine the percentage of iron in iron salt by redox titration.

Material required:-

Iron ore, potassium dichromate,HCl, $SnCl_2$, $HgCl_2$, diphenylamine indicator.

Principle:-

As an oxidant, dichromate has some advantages over permanganate, but, as it is less powerful, its use is much more limited. It is obtainable in a state of high purity and can be used as a primary standard. Solutions of dichromate in water are stable indefinitely. The half reaction for the dichromate system is:

$Cr_2O_7^{2-} + 14H^+ + 6e^- \rightarrow 2Cr^{3+} + 7H_2O \quad E° = 1.33\ V$

The most important application of dichromate is in its reaction with iron(II) in which it is often preferred to permanganate.

The relevant half reaction is :

$Fe^{2+} \rightarrow Fe^{3+} + e^- \quad E° = -0.77\ V$

and the total reaction is:

$Cr_2O_7^{2-} + 6\ Fe^{2+} + 14H^+ \rightarrow 2Cr^{3+} + 6\ Fe^{3+} + 7H_2O \quad E° = 0.56\ V$

Unlike permanganate, dichromate titrations require an indicator. There are three indicators that may be used for the titration of Fe^{2+} with $K_2Cr_2O_7$. These are diphenylamine, diphenylbenzidine and diphenylamine sulfonate. The colour change for all three indicators is green to violet and the standard electrode potentials are all ca 0.78 V.

According to Kolthoff and Sandell, this should lie between the electrode potentials of the two reduction reactions. This not being the case, phosphoric acid is added to reduce the electrode potential for the $Fe^{3+} \rightarrow Fe^{2+}$ reaction by stabilising the ferric ion.

Procedure :-

1. Weigh out accurately about 3-4 gm of dry powdered iron ore and transfer it to a 250ml beaker.

2. Add to it about 50 ml of dil. HCl .cover the beaker by a watch glass.

3. Heat the contents gently, till the ore dissolved.

4. Wash the watch-glass and the sides of the beaker and transfer the solutions to 250ml measuring flask.make the volume up to the marks.

5. Pipette out 25ml of this solution in a conical flask, add 2-3 ml of conc. HCl and heat to boiling.

6. Then, add to it SnCl2 solution dropwise, till yellow colour of solution is just discharged.

7. Add 1-2 ml of a saturated solution of HgCl2. A white turbidity is obtained.

8. Titrate the ferrous solution thus obtained against 0.1 N $K_2Cr_2O_7$ solution using indicator.

9. Repeat to get three concordant readings.

Observation table :-

S.NO.	VOLUME OF SAMPLE	BURETE READING OF $K_2Cr_2O_7$ SOLUTION		VOLUME OF $K_2Cr_2O_7$ (in ml)
		INITIAL	FINAL	

Calculation :-

Let wt. of the ore dissolved be **x**gm in 250ml solution.then, strength of ore in solution is **4x** g/lit.

Suppose 25 ml of the solution after reduction required **V** ml of 0.1 n $K_2Cr_2O_7$.

Writing normality equation for the titration, we have,

25ml of N' Fe^{2+} soln. = V ml of 0.1 N $K_2Cr_2O_7$

Applying normality formula, $N_1V_1 = N_2V_2$, we get,

25 x N' = V x 0.1
N' =V/250
Therefore, strength of iron in solution = (V/250) x 56 g/lit.

Hence, the percentage of iron in ore = 56V/250 X 100/4x

= 5.6 V/x

Precautions :-

1. The minimum is the number of drops taken out, the greater will be the accuracy.
2. Glasswares should be cleaned properly.

EXPERIMENT NO. 5

Aim:-

To determine the percentage of copper in given sample of brass iodometrically.

Material required:-

Standard sodium thiosulphate solution ($Na_2S_2O_2$), brass solution, 10% KI solution, NH_4OH, acetic acid, freshly prepared starch solution.

Principle:-

Volumetric analysis in which iodine librated during a chemical reaction from KI is determined, is known as iodometric titration.

$$CuSO_4 + 4KI \longrightarrow Cu_2I_2 + 2K_2SO4 + I_2$$

Iodine thus set free is made to react with Standard sodium thiosulphate solution ($Na_2S_2O_2$) as follows:

$$2Na_2S_2O_2 + I_2 \longrightarrow 2NaI + Na_2S_4O_6$$

By this method substance which liberate iodine from KI by chemical reaction, can be estimated by titration against Standard sodium thiosulphate solution. In iodometery, excess of KI is to be added and the estimation is always done in a conical flask or glass stoppered bottle in order to avoid loss of I_2 by evaporation. Freshly prepared starch solution is added as an indicator.

Procedure:-

1. Wash the pipette, burette and conical flask with deionized water.
2. Using the funnel, fill the burette with the Standard sodium thiosulphate solution. Open the tap briefly to fill the part below the tap. Remove the funnel. Adjust the level of the solution to the zero mark. Make sure that the burette is vertical.
3. Transfer 10 ml of given brass solution to conical flask.
4. Add ammonia solution drop by drop with constant shaking. Stop adding ammonia solution when blue ppt. formed.
5. Now add dil. Acetic acid to dissolve blue ppt.
6. Add about 25ml of 10% KI and the solution turns reddish brown.
7. Titrate immediately the iodine liberated against Standard sodium thiosulphate solution filled in burette.

8. Pale yellow color obtained then adds starch solution, blue black color was obtained.
9. Titrate with Standard sodium thiosulphate solution till blue color disappears.

Observation and calculation

S.No.	Volume of the solution of brass sample	First reading of burette	Second reading of burette	Volume of sodium thiosulphate solution
1				
2				
3				

Weight of watch glass = W1 gm

Watch glass + wt, of brass sample = W2 gm

Wt. of brass sample = (W2 –W1)

Percentage of copper in brass sample $= \dfrac{y \times at.wt. \times 100}{W1 - W2}$

Result

In the given sample of brass the quantity of copper is ………………

EXPERIMENT NO. 6

Aim – To determine the kinetic viscosity of the given sample of oil of various temperature and to study corresponding variation with respect to temperature

Apparatus required

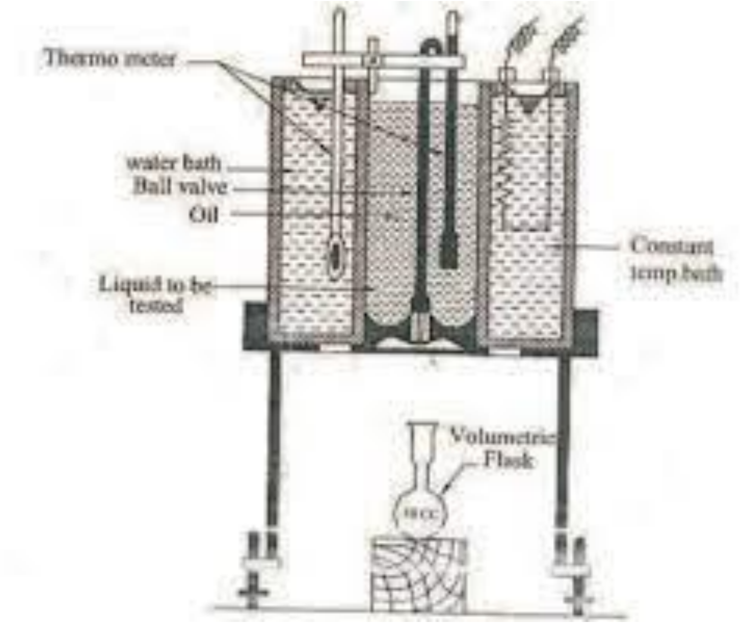
1. Red wood viscometer with accessories

2. Measuring 1 cook

3. Thermometer

4. Stop watch

Theory – Red wood viscometer is based on the principle of burner flow through the capillary tube of standard dimension under falling head the viscometer consists of vertical cylinder with on article of the center of the base of inner cylinder .The cylinder is surrounded by a water both which can maintain temperature of the liquid to be tested of the base of inner cylinder. Cylinder which is filled up to a fixed height with liquid whose viscosity is to be determined is heated by water to the desired temperature. Then open the vessel and the time required to pass the 50cc of oil is noted. With the arrangement variation of viscometer with temperature can be studies.



Procedure – The instrument was leveled with the help circular buddle and by leveling tool scraws the water are oritice is closed with the ball value and the cylinder is filled up to index mark with oil the steady state temperature of oil was recorded the procedure is repeated for different by heating oil with water both.

Formula – In this case of red wood viscometer the kinetic viscometer (v) of liquid and time (t) required to pass 50 cc of liquid are corrected by expersion

$$V = 0.0026-1.17s/t$$

Where v= kinetic viscosity and time in seconds to collect 50cc of oil

Observation -

S.no.	Volume of oil/sample	Time required	Mean time required
1			
2			
3			

Significance-

1) Pump design can be done with the help of viscosity.

2) Pump operation of the engine depends on the proper viscosity of the liquid full.

3) It is very important to know about the flow through various objects.

4) Converging of liquid can be done effects by mean of viscosity data.

Result- The kinetic viscosity for the given sample was ………………………..

EXPERIMENT NO. 7

AIM- To determine the flash and fire point of a given bituminous material.

APPARATUS: Pensky- martens closed cup tester, thermometer, heating source, flame exposure.

THERORY:-

At high temperatures depending upon the grades of bitumen materials leave out volatiles. And these volatiles catches fire which is very hazardous and therefore it is essential to qualify this temperature for each bitumen grade. BIS defined the flash point as the temperature at which the vapour of bitumen momentarily catches fire in the form of flash under specified test conditions. The fire point is defined as the lowest temperature under specified test conditions at which the bituminous material gets ignited and burns.

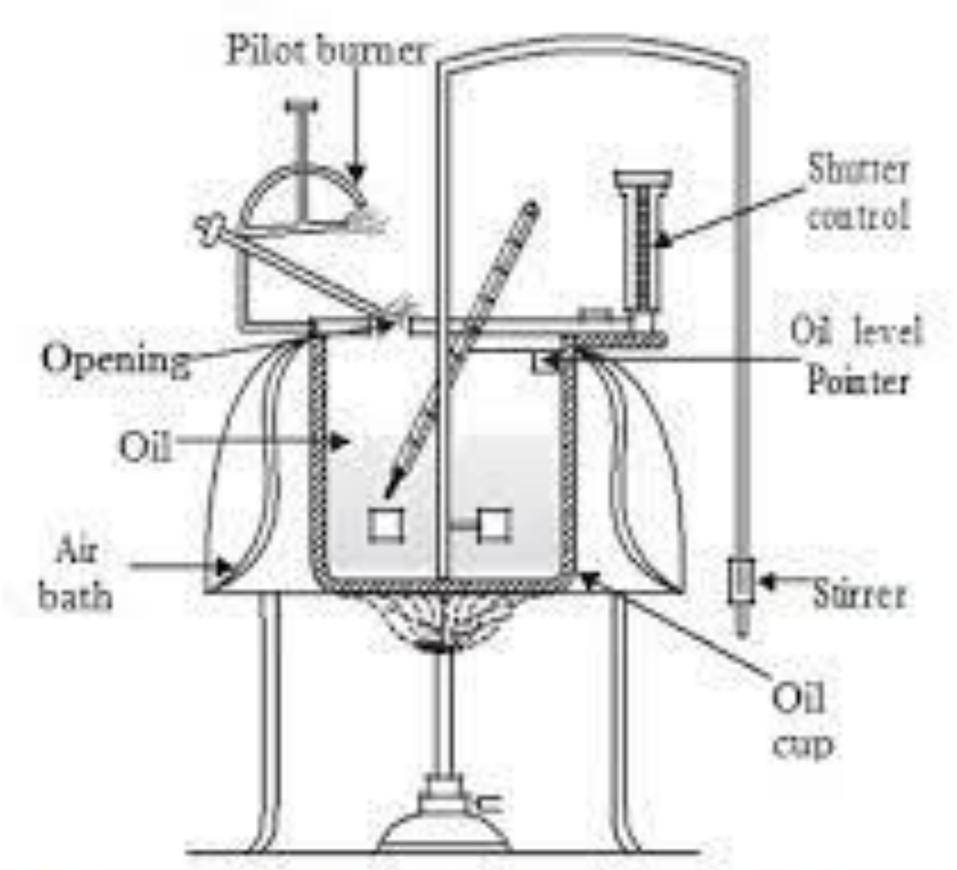


Fig. Pensky Marten's flash point apparatus

PROCEDURE:

1. All parts of the cup are cleaned and dried thoroughly before the test is started.

2. The material is filled in the cup upto a mark. The lid is placed to close the cup in a closed system. All accessories including thermometer of the specified range are suitably fixed.

3. The bitumen sample is then heated. The test flame is lit and adjusted in such a way that the size of a bed is of 4mm diameter. The heating of sample is done at a rate of 5o to 6 oC per minute. During heating the sample the stirring is done at a rate of approximately 60 revolutions per minute.

4. The test flame is applied at intervals depending upon the expected flash and fire points and corresponding temperatures at which the material shows the sign of flash and fire are noted

At high temperatures depending upon the grades of bitumen materials leave out volatiles. And these volatiles catches fire which is very hazardous and therefore it is essential to qualify this temperature for each bitumen grade.

BIS defined the flash point as the temperature at which the vapour of bitumen momentarily catches fire in the form of flash under specified test conditions. The fire point is defined as the lowest temperature under specified test conditions at which the bituminous material gets ignited and burns.

OBSERVATION AND CALCULATION:

Test	1	2	3	Mean value
Flash point				
Fire point				

RESULT:

The temperature at which the flame application that causes a bright flash ______ºC and temperature at which the sample catches fire ________ºC

EXTRA NOTES

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