

Experiment: 1
ANILINE POINT & MIXED ANILINE POINT

Aim: To determine the aniline & mixed aniline point of petroleum products and hydrocarbon solvents.

Requirements: Aniline point apparatus, Thermometer, Electric heating device, pipette etc.

Theory:

Defⁿ of Aniline point: It is the lowest temp. at which the sample is completely miscible with equal vol. of aniline.

Mixed Aniline point: The minimum equilibrium soln. temp. of a mixture of two volume of aniline, 1vol. of sample and 1vol. of n- heptane of specific purity. Aniline point of a oil gives the indication of possible deterioration of a rubber sealing, packaging etc. in contact with oil. The aromatic hydrocarbon has a tendency to dissolve certain type of rubber, so low aromatic content is desired for the oil. Lighter the paraffin content, higher is the aniline point and cetane number at the same time minimum is the knocking in the diesel fuel.

Process : Clean and dry the 'U' tube and arrange the apparatus desired. Now add 20ml. of distilled aniline and 20ml. of given sample in the 'U' tube, it will form two layers. Arrange the apparatus with stirrers in such way that liquid in 'U' tube and the paraffin of the beaker are stirred simultaneously. Switch on the heater to heat the paraffin at a controlled rate, so there is no much difference in temp. of paraffin bath and 'U' tube.

The minimum temp. at which two layers give a single phase is noted as aniline point. Now stirring of 'U' tube mixture is stopped, and mixture is allowed to cool. Temp. at which two layers are formed is also considered as aniline point.

For mixed aniline point the above procedure is repeated by taking 10ml. of sample, 10ml. of n-heptane and 20ml. of aniline.

Result: (1) Aniline point of the given sample = -----
(2) Mixed Aniline point of the given sample = -----

Observations:

(A) Aniline point:

- (1) Vol. of Aniline taken = -----
- (2) Vol. of Kerosene/ Diesel taken = -----
- (3) Cloud formation temp. = -----
- (4) One phase temp.(Aniline point) = -----
- (5) Two phase formation temp. = -----

(B) Mixed Aniline point:

- (1) Vol. of Aniline taken = -----
- (2) Vol. of n- heptane taken = -----
- (3) Vol. of Kerosene/ Diesel taken = -----
- (4) Cloud formation temp. = -----
- (5) One phase temp. (Aniline point) = -----
- (6) Two phase formation temp. = -----

Experiment: 2

DIESEL INDEX AND CETANE NUMBER

Aim: To determine the diesel index of liquid petroleum product and hydrocarbons.

Requirements: Specific gravity bottle, wt. box, thermometer, aniline point apparatus, sample oil etc.

Theory: The diesel index is indication of ignition quality of diesel fuel. Although it is of same order of the cetane number. It may differ widely from it and should therefore be accepted. The diesel index is useful for controlling ignition temp. It is calculated from aniline point of the diesel oil and specific gravity of diesel fuel at 60°F.

$$\text{Degree API} = \frac{141.5}{\text{Relative density at } 60/60^{\circ}\text{C}} - 131.5$$

$$\text{Diesel Index} = \frac{\text{Degree of API} * \text{Aniline point}}{1000}$$

Cetane number: It is a measure of ignition characteristic of diesel fuel by comparison with a range of fuel, in which cetane is given a value 100 methyl naphthalene zero.

$$\text{Cetane No.} = 0.72 \text{ Diesel Index} * 10$$

Process : Aniline point of diesel oil can be calculated from previous experiments.

Determination of specific gravity bottle of diesel: Clean & dry specific gravity bottle and determine wt. of empty bottle. Now fill the bottle with water and again wt. it. Lastly fill the bottle with diesel and weigh. Determine the wt. of diesel & water, and calculate relative density and Diesel index.

Results: Diesel index of a diesel fuel = -----

Observations:

- (1) Wt. of empty specific gravity bottle = -----
- (2) Wt. of bottle + water = -----
- (3) Wt. of bottle + Diesel = -----
- (4) Wt. of water only = -----
- (5) Wt. of diesel only = -----
- (6) Room temperature = -----

Calculation:

- (1) Relative density at room temp. = -----
- (2) Degree of API = -----
- (3) Diesel Index = -----
- (4) Cetane Number = -----

Experiment: 3

DISTILLATION

Aim: To study the distillation characteristics of light petroleum product by Distillation.

Requirements: Distilling flask, condenser, temp. controlling device, thermometer, 100ml. graduate measuring cylinder, petroleum sample.

Theory: Significance of this exp. varies from product to product. In case of crude oil distillation gives some idea of the fraction that could be collected below 300°C. If it is true B.P. (T.B.P.) distillation the T.B.P. curve reveals a lot of characteristics that are useful for the design of refinery.

Process: In a clean dry 250ml. distilling flask take exactly 100ml. of the given sample. Insert the thermometer on the neck of the flask using rubber cork. Fill the condenser box ice water mixture. Arrange the as desirable. Heat the distillation flask with a slow flame and then slowly heating device. Place a 100ml. measuring cylinder at the end of the condenser pipe. When the first drop of the distillate comes out from the tube note the temp. immediately. This is known as a initial boiling point. Observe the temp. of each 5ml. collection, till 70-80ml. of distillate comes out in the measuring cylinder. Then stop the heating and cool the apparatus at room temp. A graph is plotted between distillate collected and temp. for each 5ml. collection at different intervals.

Results:

- (1) Initial boiling point = -----°C
- (2) End boiling point = -----°C
- (3) % total recovery = -----
- (4) % recovery = -----
- (5) % residue = -----
- (6) % evaporation = -----
- (7) % loss = -----

Observations:

(A) Kerosene & Petrol

- (1) Volume of sample taken = -----
- (2) Volume of distillate = -----
- (3) Volume of distillate + residue = -----
- (4) Loss in volume distillation = -----
- (5) Initial boiling point = -----
- (6) Final boiling point = -----

(B)

Sr. Number	Sample	Collected volume	Temperature	Corrected volume
		0 ml.		
		5 ml.		
		10 ml.		
		15 ml.		
		Upto		
		75 ml.		

(C) Graph:

(1) Temp. Vs Volume collected
Temp. Vs Corrected volume

(2)

Experiment: 4

FLASH & FIRE POINT

Aim: To determine flash & fire point of liquid petroleum products by cloveland open cup method.

Requirements: Cloveland open cup apparatus, thermometer, beaker, petroleum sample.

Theory:

Flash point: It is the lowest temp. at which the oil gives off vapour that will ignite where a flame is passed over surface of the oil.

Fire point: It is the lowest temp. at which sample is ignites and continues to burn for at least 5 sec.

Flash & fire point does not indicate directly the lubricating value of the oil but these are imp. When oil is expressed to the high temp service. This test provides safe guard against decomposition and fire hazards during storage, transportation, handling and other uses.

Process: Clean and dry the brass cup. Fill the cup with the sample exactly to the mark inside the cup. Adjust the micro flame. Heat the cup slowly at a controlled rate of 3°C per minute. Pass the micro flame across the cup for every rise of 3°C of temp. Record the lowest temp. at which flame flash is observed at any point over the liquid. For determination of the fire point, continue the heating of sample at a controlled rate. Pass the test flame across the centre of cup for every degree in temp. rise.

Results:

Sr. Number	Sample	Flash point °C	Fire point °C
1	Diesel		
2	Kerosene		

Observation:

Sample	Flash point °C	Fire point °C	Mean Flash point °C	Mean Fire point °C
Diesel				
Kerosene				

Experiment: 5

CLOUD & POUR POINT

Aim: Determination of cloud and pour point of heavy liquid petroleum products.

Requirements: Cloud and pour point apparatus, thermometer, test tube, petroleum sample.

Theory:

Cloud Point: It is the temp. at which a cloud or haze of wax crystal appears at the bottom of the test jar when the oil is cooled under prescribed condition.

When the oil is cooled at a specified rate, the temp. at which it becomes cloudy in appearance is formed as cloud point. This is due to the separation of the wax crystals present in dissolved form in it. It gives a rough idea of the temp. above which the oil can be safely handled without any fear. The cloud point is not so imp. for lubricating oil, But it is imp. for the fuel oil.

Process: Clean and dry the test tubes and fill it with heavy petroleum product to minimum required level. Heat the test tube on a burner to get clear & uniform consistency. Seal the test tube with a cork having thermometer of required range, the bulb of thermometer should be dipped in the sample. Now place the test tube in the ice bath in vertical position. At an interval of 2°C minimum decreasing observe the cloudiness at the bottom of test tube, bringing it out in vertical position from the apparatus. Note down the temp. at which cloud or crystals are observed. Report it as cloud point of the sample.

Continue the cooling of test tube in the same manner until temp. reaches at which the sample in test tube does not show any movement for at least 5 sec, when brought to the horizontal position. Note this as pour point of the sample.

Results:

Sample	Cloud point °C	Pour point °C
1		
2		

Observation Table:

Sample Number	Cloud point °C	Mean °C	Pour point °C	Mean °C
1 (a) (b) (c)				
2 (a) (b) (c)				
3 (a) (b) (c)				
4 (a) (b) (c)				

Experiment: 6

CARBON RESIDUE

Aim: Determination of % carbon residue of liquid petroleum products by conradson carbon residue apparatus.

Requirements: conradson carbon residue apparatus, wt. box, petroleum sample.

Theory: It serves as a rough estimation of tendency of fuel to form deposit in vaporizing pot type and slem type burner, combustion chamber; carbon residue of motor oil is regarded as amount of carbonaceous deposit in combustion chamber of engine. Carbon residue of gas oil is useful as a guide in mfg. of gas from gas oil.

Process: Weigh accurately about 1-3gms of the given sample in a clean and dry previously weighted silica crucible. Place two glass beads (2.5mm dia) in the crucible. Place in the centre of skidmore. Level the sand in the large sheet iron crucible and set skidmore crucible exactly in the centre. Cover both the iron crucible, to allow free exit to the vapor as formed.

Next centre the iron crucible in the insulator with its bottom resting on top of the triangular cover with iron hood. Apply heat with high strong flame for 10min. when smoke appears above the chimney, till the burner to ignite the vapors on the sides of the crucible. When flame is no more from the chimney, apply strong heat to flame lowest part of iron crucible for about 10min. Remove the burner and allow the apparatus to cool. Remove silica crucible, place in desiccator. Cool and weigh. Calculate % of carbon residue on original sample.

Results:

- (1) Wt. of crucible = ----- gms.
- (2) Wt. of crucible + oil = ----- gms.
- (3) Wt. of crucible + residue = ----- gms.
- (4) Wt. of oil = ----- gms
- (5) Wt. of residue = ----- gms.

$$\% \text{ carbon} = \frac{Y}{X} * 100$$

Experiment: 7

SMOKE POINT

Aim: Determination of smoke point of light petroleum products.

Requirements: Smoke point apparatus, beaker, petroleum sample.

Theory:

Smoke Point: It is the max. flame height in mm at which the sample burns without smoke.

Smoke point is related with the aromatic content of the liquid and it is inversely proportional to the aromatic content. Smoke point is used to determination of smoking tendency. Smoking tendency is proportional to the aromatic content and is given by Eq.

Smoking tendency = $320 / \text{smoke point in mm}$.

Process: Clean the sample container with suitable solvent and dry it. Fill the sample container up to desired level and introduce a wick in the container. Place this assembly in the burning chamber of the apparatus. Open the glass door, light the flame and closed the glass door. By adjustment of sliding screw observe the condition and height of the flame. If it is giving smoke, reduce the flame height to stage I, then a further to stage II and also familiarised with the flame height decrease to IIIrd stage. Adjust the point of the flame so that it burns without smoke. Note this height in mm using scale on the back side. Repeat it to get a constant reading.

Results:

Sr. Number	Sample	Smoke point	Smoking tendency
1	Kerosene		
2	Diesel		

Observation:

Sr. Number	Sample	Smoke point	Smoking tendency
1	Kerosene		
2	Diesel		

Experiment: 8

REDWOOD VISCOMETER

Aim: To determine the kinematic viscosity of given petroleum product by redwood viscometer.

Apparatus: Redwood viscometer, stop-watch, 50 ml. collection flask, oil sample, paraffin oil.

Theory: Viscosity is the resistance to flow in oil. It is defined as "force required moving a plane 1 Cm² in area at a speed of 1cm/sec. over another fixed plane separated by 1 cm. thin layer of liquid. The kinematic viscosity is the ratio of absolute viscosity to the density of liquid. Viscosity has the unit Poise or 1dyen/sec/cm².

In commercial viscometer a fixed volume of liquid is allowed to flow through standard capillary and flow time is noted. Viscosity of oil decreases with increase in temperature as it reduces the inter molecular interaction. Change in viscosity with change in temperature is indicated the viscosity index.

Process: Clean and dry the oil cylinder. Make sure that cylinder orifice is clean. Fill the sample oil upto the mark. Paraffin oil is filled in the heating bath. Using the regulator heat the oil to the desired temperature. Keeping a narrow gap between sample and oil bath temperature. A 50 ml. measuring flask is placed exactly below the jet and stopper is removed at experiment temperature.

The time for 50 ml. oil collection is noted & stopper is placed back to prevent oil overflow. Repeat the process to obtain the constant reading.

Results:

Sr. Number	Temp. of oil bath	Time in sec.	Kinematic viscosity
1			
2			
3			
4			

Observation:

- (A)
(1) Temp. of paraffin bath = ----- °C
(2) Temp. of oil bath = ----- °C

Sr. Number	Vol. collected ml.	Time in sec.
I	50	
II	50	
III	50	

Plot the graph of flow time Vs K.V.

Sr. Number	Time in sec.	K.V. Centi stock
1	106.4	22
2	115	24
3	123.7	26
4	132.5	28
5	141.3	30
6	150.2	32.0
7	186.0	40
8	195.3	42
9	204.4	44
10	213.7	46
11	222.9	48
12	232.1	50

Experiment: 9

SAYBOLT'S VISCOMETER

Aim: To determine the kinematic viscosity of oil sample using Saybolt's viscometer.

Requirements: Say bolt's viscometer, stop-watch, 60 ml. collection flask, thermometer, oil sample. Paraffin oil etc.

Process: Clean and dry the oil cylinder of viscometer. Insert the cork tightly enough to prevent any air escape. Fill the cylinder upto the mark with oil. Take out excess oil with the help of pipette. Insert a thermometer both in a oil bah and the paraffin bath. Start heating at a controlled rate and stir the oil and paraffin bath continuously for uniform heating. As the oil sample is at desired temperature open the cork at bottom and note down the time required to fill 60 ml. of oil. Repeat the process for constant reading. Find out a constant flow time at difference temperature.

Results:

Sr. Number	Temp. of oil bath	Time in sec.	Kinematic viscosity
1			
2			
3			
4			

Observation:

(A)

(1) Temp. of paraffin bath = ----- °C

(2) Temp. of oil bath = ----- °C

Sr. Number	Vol. collected ml.	Time in sec.
I	60	
II	60	
III	60	

Plot the graph of K.V. Vs Flow time.

Sr. Number	Time in sec.	K.V. Centi stock
1	106.4	22
2	115	24
3	123.7	26
4	132.5	28
5	141.3	30
6	150.2	32.0
7	186.0	40
8	195.3	42
9	204.4	44
10	213.7	46
11	222.9	48
12	232.1	50

Experiment: 10

MOISTURE CONTENT

Aim: To determine the %age of moisture present in the given sample of liquid petroleum by Dean and Stark's method.

Requirements: Dean & Stark's apparatus, sample fuel etc.

Process: Clean and dry the round bottom flask. Take exactly 100 ml. of the given sample in the flask. Arrange the apparatus; circulate the cold water in condenser. Heat the content of the flask over a small heating rate. A mixed vapour of petroleum and moisture is collected in a specified designed graduated distillation receiver after condensation. Heat the flask until a constant height of liquid is collected. Note the height of lower layer as water. The volume of water collected gives directly the results in %age moisture in the given sample.

Results: The %age moisture content in the given sample is _____%.

Observation:

(1) Vol. of petroleum sample taken = ----- ml
Vol. of water collected = -----ml. (2)