Bharati Vidyapeeth's Institute of Technology

Navi Mumbai

Certificate

This is to certify that, Mr/ Ms. …….

Roll No. …… of sixth Semester of Diploma in Chemical engineering of Baharati Vidyapeeth Institute of Technology Navi Mumbai (Inst.code:0027) has satisfactorily completed the term work in the subject Petroleum &Petrochemical Technology (22611) for the academic year 20…. to 20…. as prescribed in the MSBTE curriculum.

Place: ……... Enrollment No. : ……

Date:……. Exam. Seat No. : …….

Sign:

Name:

Subject Teacher Head of the Department Principal

Seal of Institution

List of experiments and progressive assessment for term work (TW) D-3

Academic Year: **Name of Faculty:**

Course code: CH6I **Subject Code: PPT (22611)**

Name of candidate: **Enroll no.** **Roll no.**

Semester: SIXTH **Marks: Max : 25 Min :10**

Sr. No.	Title	Date of performance	Date of submission	Marks	Sign of teacher
1	Use the specific gravity bottle to measure API gravity of given refinery product.				
2	Use the Pensky Marten's apparatus to determine flash point of kerosene				
3	Use the Pensky Marten's apparatus to measure fire point of kerosene.				
4	Use the Abel's apparatus to measure flash and fire point of petrol.				
5	Use the cloud and pour point apparatus to determine cloud and pour point of given oil.				
6	Use the drop point apparatus to measure drop point of given sample.				
7	Use the smoke point apparatus to measure smoke point of kerosene.				
8	Use the aniline point apparatus to determine aniline point of given sample				
9	Use aniline point apparatus and gravity bottle to measure diesel index of given sample.				
10	Use the Conradson apparatus to determine carbon residue of given oil sample.				
11	Use the ASTM distillation set up to measure initial and final boiling point of given petroleum product.				

12	**Use the Draw distillation characteristic curve for given sample using ASTM distillation setup.**				
13	**Use the Esterification process to prepare Ethyl Acetate.**				
14	**Use the transesterification process to prepare biodiesel from used oil.**				
15	**Prepare Phenol formaldehyde resin using Phenol.**				
Total marks out of 100					
Marks out of 25					

EXPERIMENT: -1

Aim: Use the specific gravity bottle to measure API gravity of given refinery product.

Theory: Specific gravity of oil is often expressed as degree API. API gravity (American Petroleum Institute gravity) is a measure of how heavy or light petroleum liquid is compared to water. If its API gravity is greater than 10, it is lighter and floats on water, if less than 10, it is heavier and sinks.

Deg API = (141.5 / Specific gravity at 15.56^0C) – 131.5

Specific gravity is an indication of the type of hydrocarbon present; being highest for aromatics and lowest for paraffin. The API gravity reverses the relationship. API gravity is used to compare the densities of petroleum liquids. API gravity of most petroleum liquids fall between 10 and 70 degrees.

Procedure:

1. Weigh an empty specific gravity bottle with stopper of known volume(W_1).
2. Fill the specific gravity bottle with the given oil (lubricating oil) and place the stopper.
3. Note the weight of the filled bottle with stopper (W_2).
4. Find out the specific gravity and API gravity.

Calculation:

a. Volume of specific gravity bottle = ……ml

b. Weight of empty bottle with stopper (W_1) = …….g.

c. Weight of bottle + oil (W_2) = …….g

d. Weight of oil = W_2 - W_1 = ……

e. Density of oil = Weight of oil / volume of specific gravity bottle = ……..

f. Specific gravity of oil = Density of oil / density of water = ……

g. Deg API = (141.5 / Specific gravity at 15.56^0C) – 131.5 = ……

Result:

API gravity of given oil is ……..

Questions:

1. Define API gravity.
2. Give the formula to calculate API gravity of a mixture of close boiling cuts or a blend.

3. Give the significance of API gravity.

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C(4)	P(4)	A(2)	TOTAL	SIGN

EXPERIMENT: -2

Aim: - Use the Pensky Marten's apparatus to determine flash point of kerosene.

Apparatus required:: - Pensky Marten's flash and fire point apparatus, match box, thermometer,

Description of Pensky Marten's apparatus

It is used to determine the flash point of the lubricating oils, fuel oils, solvents, solvent containing material and suspension of solids. It consists of three parts

a) Oil Cup

Material- Brass

Height – 5.5cm

Diameter-5cm

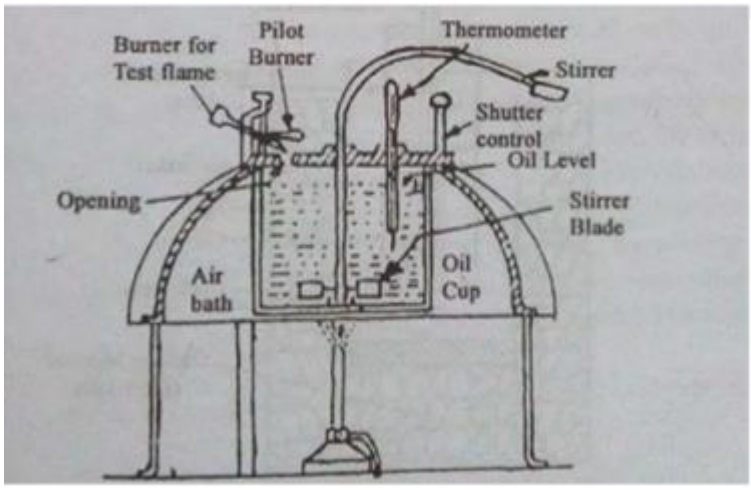
Lid of the cup is provided with four openings of standard sizes, first opening is for stirrer, second is for admission of air, third is for thermometer and fourth is for introducing test flame

b) Shutter

At the top of the cup shutter is provided. By moving the shutter, opening in the lid opens and flame is dipped in to this opening, bringing the flame over the oil surface. As the test flame is introduced in the opening, it get extinguished, but when the test flame is returned to its original position, it is automatically lightened by the pilot burner.

c) Stove:

It consists of 1. Air bath, 2. Top plate on which the flange of the cup rest



Principle: -

Flash point is the lowest temperature at which the lubricating oil gives off enough vapors that ignite for a moment when tiny flame is brought near it.

Flash and fire points are used to indicate

a. Fire hazard of petroleum products and evaporation loses under high temperature.
b. It gives us the idea about the maximum temperature below which the oil can be used
c. It is used as the means of identification of specific lubricating oil
d. For detection of contamination in the given oil

Procedure: -

1. Clean and dry all parts of the apparatus with the help of suitable solvent e.g. CCl4, ether, petroleum spirit or benzene and dry it to remove any traces of solvent.
2. Fill the oil cup with the test oil up to the mark.
3. Fix the lids on the top through which are inserted a thermometer and a stirrer. Ensure that the flame exposure device is fixed on the top.
4. Light the test flame and adjust it to about 4 mm in diameter.
5. Heat the apparatus as temperature of oil increases by 5 to 60 per min. as stirrer is continuously rotated.
6. At every 1^0 C rise of temperature, introduce test flame into the oil vapor. This is done by operating the shutter. On moving knob of shutter, test flame is lowered in oil vapors through opening.
7. When test flame causes a distinct flame in interior cup, note temperature which represent the flash point

Result: - The flash point of given oil sample=……..^{0} C

Questions:

1) What is flash point?

2) Explain the significance of flash point.

Answers:

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C(4)	P(4)	A(2)	TOTAL	SIGN

EXPERIMENT: -3

Aim: - Use the Pensky Marten's apparatus to measure fire point of kerosene.

Appararus required: - Pensky Marten's flash and fire point apparatus, match box, thermometer.

Description of Pensky Marten's apparatus

It is used to determine the fire point of the lubricating oils, fuel oils, solvents, solvent containing material and suspension of solids.

It consists of three parts

a) Oil Cup

Material- Brass

Height – 5.5cm

Diameter-5cm

Lid of the cup is provided with four openings of standard sizes, first opening is for stirrer, second is for admission of air, third is for thermometer and fourth is for introducing test flame

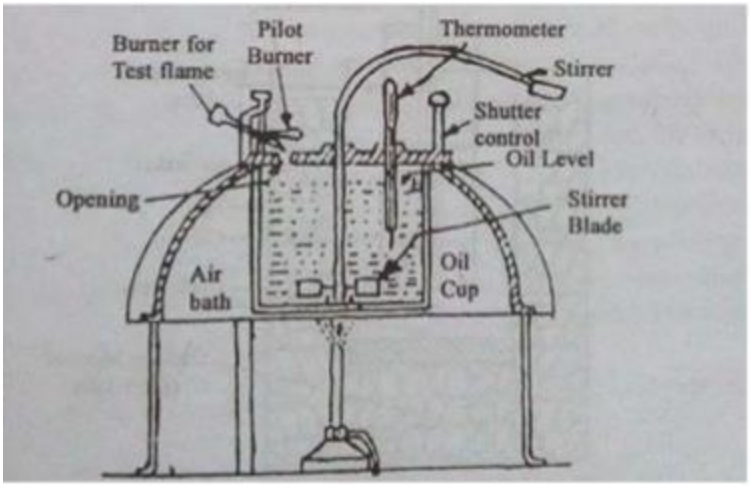
b) Shutter

At the top of the cup shutter is provided. By moving the shutter, opening in the lid opens and flame is dipped in to this opening, bringing the flame over the oil surface. As the test flame is introduced in the opening, it get extinguished, but when the test flame is returned to its original position, it is automatically lightened by the pilot burner

c) Stove

It consists of 1. Air bath, 2. Top plate on which the flange of the cup rest

Pensky Marten's (closed cup) flash point apparatus



Principle: -

Flash point is the lowest temperature at which the lubricating oil gives off enough vapors that ignite for a moment when tiny flame is brought near it.

Fire point is the lowest temperature at which the vapors of the oil burn continuously for at least five seconds when a tiny flame is brought near it.

Flash and fire points are used to indicate

a. Fire hazard of petroleum products and evaporation loses under high temperature loses.
b. It gives us the idea about the maximum temperature below which the oil can be used.

c. It is used as the means of identification of specific lubricating oil.
d. For detection of contamination in the given lubricating oil.

Procedure: -

1. Clean and dry all parts of the apparatus with the help of suitable solvent e.g. CCl4, ether, petroleum spirit or benzene and dry it to remove any traces of solvent.
2. Fill the oil cup with the test oil up to the mark.
3. Fix the lids on the top through which are inserted a thermometer and a stirrer. Ensure that the flame exposure device is fixed on the top.
4. Light the test flame and adjust it to about 4 mm in diameter.
5. Heat apparatus as temp. of oil increases by 5 to 6^0 per min. as stirrer is continuously rotated.
6. At every 1^0 C rise of temp. Introduce test flame into the oil vapor. This is done by operating the shutter. On moving knob of shutter, test flame is lowered in oil vapors through opening.
7. When test flame causes a distinct flame in interior cup, note temp. which represent the flash point
8. Further heat the oil at the rate of 1^0C/ min. and continue applying the test flame as before.
9. The temperature at which the vapors of the oil give a clear and distinct blue flash for five seconds is recorded as the fire point of the oil.

Result: -

The fire point of given oil sample=____ C.

Questions:

1) What is flash point?
2) What is fire point?
3) Explain the significance of flash& fire point.

Answers:

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C(4)	P(4)	A(2)	TOTAL	SIGN

EXPERIMENT 4

Aim: Use the Abel's apparatus to measure flash and fire point of petrol.

Apparatus:

Abel's apparatus, Thermo meter (0-110^{o}C).

Theory:

This method determines the closed cup flash and fire points of petroleum products and mixtures to ascertain whether they give off inflammable vapours below a certain temperature.

Flash point:

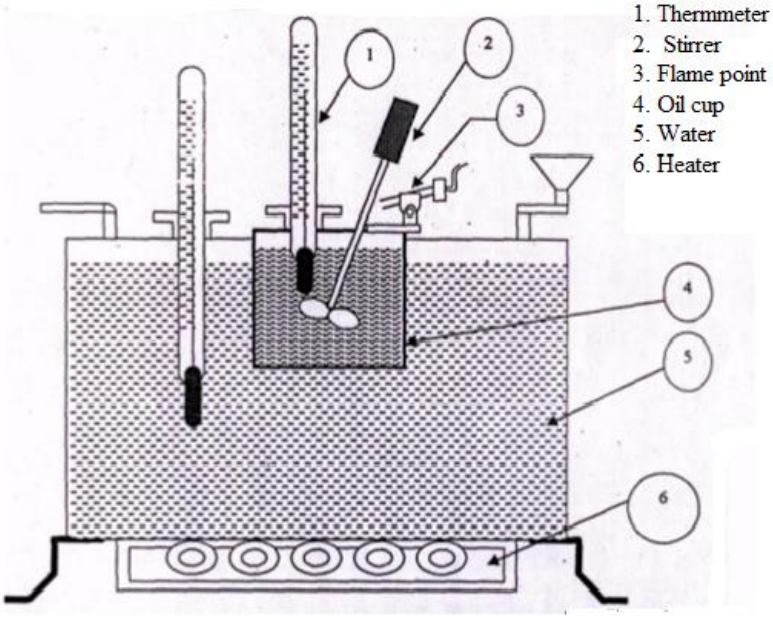
It is the lowest temperatures of the oil, at which, application of test flame causes the vapour above the sample to ignite with a distinct flash inside the cup.

Fire point:

It is the lowest temperature of the oil, at which, application of test flame causes burning for a period of about five seconds.

Description:

The apparatus consists of a brass cup and cover fitted with shutter mechanism, test flame arrangement, hand stirrer, thermometer socket. The brass cup is heated by water bath (with energy regulator), fitted with a funnel and overflow pipe.



Procedure

1. Clean the oil cup and fill the up to the mark with the petrol.
2. Insert the thermometer into the oil cup through the provision to note down the temperature of petrol
3. Using the Energy regulator, control the power supply given to the heater and rate of heating
4. The petrol is heated slowly when temperature of petrol rises; it is checked for the flash point

for every one-degree rise in temperature.

5. After determining the flash point, the heating shall be further continued. The temperature at which time of flame application that causes burning for a period at least 5 seconds shall be recorded as the fire point.

Result:

1. Flash point of petrol is …….^{0}C.

2. Fire point of petrol is ……^{0}C

Questions:

1. Give the standard value of flash point for diesel, petrol, kerosene.

2. Explain the working of Abel's apparatus.

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C(4)	P(4)	A(2)	TOTAL	SIGN

EXPERIMENT 5

Aim : Use the cloud and pour point apparatus to determine cloud and pour point of given oil.

Apparatus: Cloud & pour point apparatus, thermometer

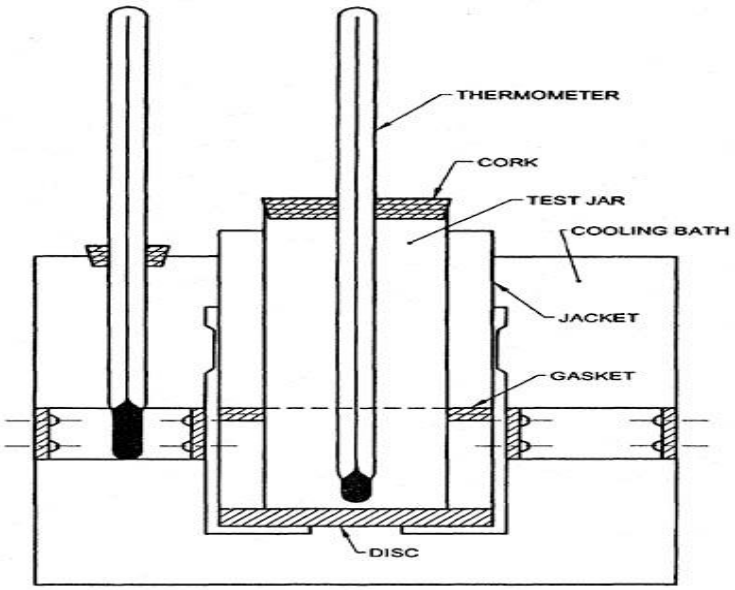
Theory:

Cloud point: The temperature, at which a cloud or haze appear when the oil is cooled under prescribed conditions.

Pour point: The temperature at which oil ceases to flow when it is cooled is known as pour point.

Cloud and pour points are related to low temperature characteristics of fuel and tells the behaviour of fuel at low temperatures. Haziness may be due to separation of waxes or increase in viscosity at lower temperature. Due to the separation of waxes, viscosity of fuel increase and its fluidity fluidity decreases. The presence of solidified waxes thickens the oil and clogs fuel filters and injectors in engine.so the presence of these solid waxes affects the performance of engines. Usually the difference between fash and fire point is 4-6^{0}F. Pour point tells us the temperature below which oil cannot be used as lubricant. Cloud point indicates the tendency of oil to plug filters or small orifices at cold operating temperatures. So cloud and pour points tell us the suitability of oil in cold weather conditions.

Cloud & pour point apparatus consists of a test jar with provision for introducing thermometer and cork for sealing it tightly. Thermometer and jar are coaxial. A ring gasket is placed around the test jar at a height of one inch from the bottom. The test jar is placed in a jacket. The whole assembly of jacket with test jar is placed in the cooling bath.



Procedure:

1. Bring the sample to be tested (clear oil) to a temperature of at least 15^0C above the approximate cloud point.
2. Pour the clear oil in to the test jar up to the level marked.
3. Adjust the position of the cork carrying test thermometer so that the cork fits tightly.
4. Insert the test jar in the jacket and put this jacket in the cooling bath maintained at a lower temperature.
5. At every 2^0C fall in temperature, remove the jar from the jacket quickly but without disturbing the oil, inspect the material for cloud, and replace the jar; this complete operation shall not take more than 3 seconds.
6. Repeat the procedure till distinct cloudiness or haze appears at the bottom of the jar, record the reading of the thermometer as the cloud point.
7. After determining cloud point, again cool the oil sample.
8. At every 2^0C fall in temperature, remove the jar from the jacket carefully, and tilt it just enough to see whether the oil will move and then replace it, this complete operation shall not take more than 3 seconds.
9. Repeat the procedure till the sample ceases to flow when the jar is tilted. Then hold the jar in horizontal position for exactly 5 seconds. If the oil shows no movement during the 5 seconds, record the reading of the thermometer as the pour point.

Result:

Cloud point of given sample is ……..^{0}C
Pour point of given sample is ……^{0}C

Questions:

1. Define cloud point and pour point.
2. Explain the construction of the cloud and pour point apparatus.

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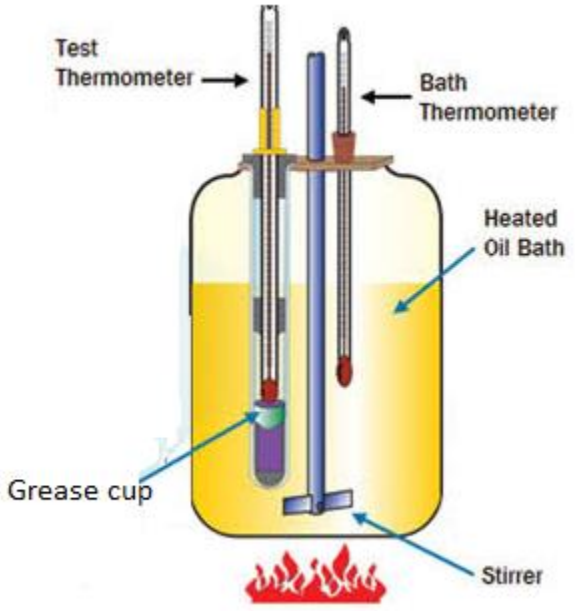
C(4)	P(4)	A(2)	TOTAL	SIGN

EXPERIMENT 6

Aim: Determination of Drop Point.

Apparatus: Drop point apparatus, thermometer.

Theory: Drop point determinations are used for identification and quality control purposes, and can be an indication of the highest temperature of utility for some applications. The sample is heated at a prescribed rate in a precision machined cup whose sides slope toward an opening at its center. The temperature at which a liquid drop first falls from the cup is the dropping point of the sample. The dropping point of lubricating grease is an indication of the heat resistance of the grease and is the temperature at which it passes from a semi-solid to a liquid state under specific test conditions. It is dependent on the type of thickener used and the cohesiveness of the oil and thickener of grease. The dropping point indicates the upper temperature limit at which a grease retains its structure though is not necessarily the maximum temperature at which a grease can be used. Dropping point is used in combination with other testable properties to determine the suitability of greases for specific applications and for use in quality control.



Procedure

1. The oil/block is heated, while being stirred; at a rate of 4°C to 6°C per minute until the temperature is approximately 17 °C below the expected dropping point. The heat is reduced until the test tube temperature is at most 2.2 °C less than the oil/block temperature.
2. Once the temperature has stabilized the sample is inserted.
3. Heating is continued till the drop emerges from the cup.
4. The drop point is the temperature recorded on the test tube thermometer when a drop of grease falls through the hole in the grease cup. If the drop trails a thread, the dropping temperature is the temperature at which the thread breaks

Result:

Drop point of grease is …….^{0}C

Questions:

1. Define octane number?
2. Define drop point?
3. Define ignition temperature?

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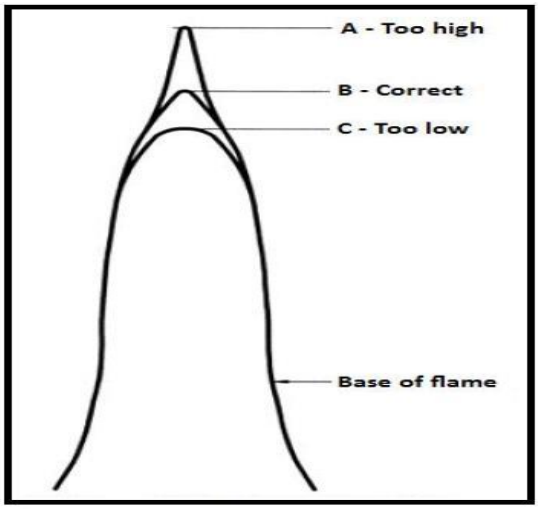
C(4)	P(4)	A(2)	TOTAL	SIGN

EXPERIMENT 7

Aim: Use the smoke point apparatus to measure smoke point of kerosene.

Apparatus: smoke point apparatus, wick

Theory: It is a measure of the tendency of a liquid fuel to produce carbon particles known as soot. Generally, it is measured by burning fuel in a special wick lamp in which the flame height is increased slowly till it starts producing smoke. The maximum height in term of millimeters of smokeless flame at which flame starts smoking is termed as smoke point. Hence, higher the smoke point, lower will be the tendency of the fuel to 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. This test method provides an indication of the relative smoke producing properties of kerosene. The smoke point is related to the hydrocarbon type composition of such fuels. Generally, the more aromatic fuel the smokier the flame. The smoke point is quantitatively related to the potential radiant heat transfer from the combustion products of the fuel.



Procedure:

1. Soak a piece of extracted and dried wick (about 125 mm) long in the sample (Kerosene). Place it in the wick tube of candle.
2. Fill the sample container up to desired level (20 ml) and introduce a wick in the container.
3. Cut the wick horizontally (6 mm) from the end of the candle.
4. Place this assembly in the burning chamber of the device.
5. Open the glass door, light the flame and adjust the wick (The flame should be about 10mm height). Allow the lamp to burn for 5 min.
6. Raise the candle until smoke appears from from the chimney (Stock).
7. Slowly lower the candle until the smoke disappear.
8. Take the reading from the reflection of the flame image on the scale. This reading represents smoke point of the sample.
9. It is quite recommended that to take more than one observation to get right reading.

Result:

Smoke point of kerosene is ……mm

Questions:

1. What is the significance of smoke point test?
2. Draw typical flame appearance

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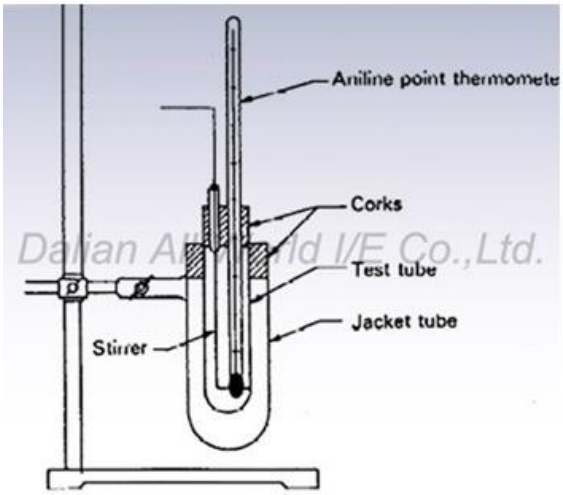
C(4)	P(4)	A(2)	TOTAL	SIGN

EXPERIMENT -8

Aim : Use the aniline point apparatus to determine aniline point of given sample.

Theory: The aniline point of oil is defined as the minimum temperature at which equal volumes of aniline and oil are miscible. The value gives an approximation for the content of aromatic compounds in the oil, since the miscibility of aniline, which is also an aromatic compound suggests the presence of similar (i.e. aromatic) compounds in the oil. The lower the aniline point, the greater is the content of aromatic compounds in the oil as obviously a lower temperature is needed to ensure miscibility. The aniline point serves as a reasonable proxy for aromaticity of oils consisting mostly of saturated hydrocarbons (i.e. alkanes, paraffins) or unsaturated compounds (mostly aromatics). Significant chemical functionalization of the oil (chlorination, sulfonation, etc.) can interfere with the measurement, due to changes to the solvency of the functionalized oil.



Procedure:

1. Clean and dry the apparatus.
2. Deliver 10 ml of aniline and 10 ml of the dried sample into the test tube fitted with stirrer and thermometer. If the material is too viscous for volumetric transfer, weigh to the nearest 0.01 g a quantity of the sample corresponding to 10 ml at room temperature.
3. Center the thermometer in the test tube so that the immersion mark is at the liquid level, making sure that the thermometer bulb does not touch the side of the tube.
4. Center the test tube in the jacket tube.
5. Stir the mixture rapidly using a 50-mm (2-in.) stroke, avoiding the introduction of air bubbles.
6. If the aniline-sample mixture is not miscible at room temperature, apply heat directly to the jacket tube so that the temperature rises at a rate of 1 to 3°C /min by removing or reducing the heat source until complete miscibility is obtained.
7. Continue stirring and allow the mixture to cool at a rate of 0.5 to 1.0°C min.

8. Continue cooling to a temperature of 1 to 2°C below the first appearance of turbidity, and record as the aniline point the temperature at which the mixture suddenly becomes cloudy throughout.

Result: Aniline point of given sample is …….^{0}C

QUESTIONS:

1) Define aniline point?

2) Give the significance of aniline point.

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C(4)	P(4)	A(2)	TOTAL	SIGN

EXPERIMENT -9

Aim : Use aniline point apparatus and gravity bottle to measure diesel index of given sample.

Theory: Diesel fuels are mainly composed of paraffin although there is no bar for aromatics. Aromatics of this boiling range present in the fuel cause abnormal ignition delay. For this reason, an estimation of aromatics is essential. One such method is aniline point which can predict the suitability of oil. A low aniline point indicates low diesel index because of high percentage of aromatics. Diesel index is a measure of ignition quality of fuel. Diesel index or cetane number refers to the ease with which diesel fuel ignites easily at a relatively lower temperature. Self-ignition temperature is low for paraffin while it is high for aromatics. Thus a fuel rich in aromatics burns later causing ignition delay and it gives rise to what is known as diesel knock. A high diesel index is also not desirable as a fuel rich in aromatics give rise to better calorific value than paraffin rich fuel of equal weights.

Procedure:

1. Clean and dry the aniline apparatus.
2. Deliver 10 ml of aniline and 10 ml of the dried sample into the test tube fitted with stirrer and thermometer. If the material is too viscous for volumetric transfer, weigh to the nearest 0.01 g a quantity of the sample corresponding to 10 ml at room temperature.
3. Center the thermometer in the test tube so that the immersion mark is at the liquid level, making sure that the thermometer bulb does not touch the side of the tube.
4. Center the test tube in the jacket tube.
5. Stir the mixture rapidly using a 50-mm (2-in.) stroke, avoiding the introduction of air bubbles.
6. If the aniline-sample mixture is not miscible at room temperature, apply heat directly to the jacket tube so that the temperature rises at a rate of 1 to 3°C /min by removing or reducing the heat source until complete miscibility is obtained.
7. Continue stirring and allow the mixture to cool at a rate of 0.5 to 1.0°C min.
8. Continue cooling to a temperature of 1 to 2°C below the first appearance of turbidity, and record as the aniline point the temperature at which the mixture suddenly becomes cloudy throughout.
9. Weigh an empty specific gravity bottle with stopper of known volume(W_1).
10. Fill the specific gravity bottle with the diesel oil and place the stopper.
11. Note the weight of the filled bottle with stopper (W_2).
12. Find out the specific gravity and API gravity.

Calculation:

a. Aniline point of diesel =

b. Volume of specific gravity bottle = ……ml

c. Weight of empty bottle with stopper (W_1) = …….g.

d. Weight of bottle + diesel oil (W_2) = …….g

e. Weight of diesel oil = $W_2 - W_1$ = ……

f. Density of diesel oil = Weight of diesel oil / volume of specific gravity bottle =

g. Specific gravity of diesel oil = Density of diesel oil / density of water

= ……

h. Deg API = (141.5 / Specific gravity at 15.56^0C) – 131.5 = ……

i. Diesel index = (0.018 *aniline point ^{0}C +0.32) API gravity

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Result:

Diesel index =

Questions:

1. Define cetane number.

2, Explain the significance of diesel index.

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C(4)	P(4)	A(2)	TOTAL	SIGN

EXPERIMENT 10

Aim: Use the Conradson apparatus to determine carbon residue of given oil sample.

Apparatus: a) Porcelain crucible, b) Iron Crucible c) spun sheet iron crucible d) wire support e) Hood f) insulator and g) burner.

Theory:

Oils contain mainly chemical compounds of carbon and hydrogen. If heated in a closed vessel in the absence of sufficient air, the oil will vaporise and a thin deposit of carbon residue will be left behind. This test serves as index and gives some relative measure of the amount of residue to be formed by lubricating oils, especially in IC Engines.

Conradson Carbon residue tester consists of a glazed porcelain crucible of about 25 ml capacity, placed in iron crucible of 80 ml approximate capacity and having cover with an opening of 5 to 6 mm dia. The two together are placed in a spun sheet iron crucible with cover, about 200 ml capacity at the bottom of which is placed a layer of about 25g clean dry sand such that the iron crucible containing the glazed porcelain crucible with its cover on it is brought to the top of the sheet iron crucible. The spun sheet iron crucible is supported by a triangular base at the bottom. A circular sheet iron hood with a sloping top and provided with central chimney, encloses the crucible.

Procedure:

1. The crucible is weighed accurately.
2. About 10 gms of oil to be tested for carbon residue is taken in the crucible and weighed.
3. This crucible is placed in the centre of the iron crucible.
4. Now sand is taken in the spun sheet iron crucible and is evenly spread and on the sand bed the iron crucible with the glazed porcelain crucible containing the oil sample is placed.
5. The whole assembly is placed in the sheet iron block and the cover is put in position.
6. The hood is placed on the block and heat is applied with the burner at the bottom of the spun iron crucible.
7. After about 20 to 25 minutes of heating, the cover is slightly displaced to make the vapour escape, simultaneously introducing a lighted splinter in the hood.
8. If sufficient vapours have formed, they will catch fire and continue to burn.
9. When the flame dies down, it means all the oil taken has been vaporized.
10. The hood is removed first, then, the cover is lifted, the crucible is taken out and placed in a desicator for getting cooled.
11. After the crucible is cooled down to ambient temperature, it is weighed again.
12. The difference between the initial and final weights of the crucible give the amount of carbon residue and is expressed as a percentage of carbon residue as shown in the calculation.

NOTE: For sample having carbon residue from 5 to 10%, sample of 5+0.5 gm is to be taken. If the carbon residue exceeds 15%, a sample of 3 + 0.1 gm should be taken.

Calculations:

a. Weight of crucible with glass beads =W_1 = ……..g.
b. Weight of crucible with oil =W_2 = …….g
c. Weight of oil taken = $W_0 = W_2 - W_1$ = ……..g
d. Weight of the crucible and residue = W_3 = ……g
e. Weight of the carbon residue = Wc = $W_3 - W_1$
f. Percentage of carbon residue = Wc/Wo * 100

Result:

The percentage of carbon present in given sample of lubricating oil is ……. %

Questions:

1. Define carbon residue
2. Explain the working of Conradson apparatus

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C(4)	P(4)	A(2)	TOTAL	SIGN

EXPERIMENT 11

Aim: Use the ASTM distillation set up to measure initial and final boiling point of given petroleum product.

Theory:

ASTM distillation refers to a diverse group of different international standards used for determining the volatility characteristics of petroleum products. ASTM distillation specifies the evaporation characteristics of gasoline. Ease of starting is governed by IBP to 10% range boiling point. Lower IBP's are preferred for cold climates. A drawback of very low IBP is vapour locking of the engine due to high evaporation.

Procedure:

1. 100 ml of gasoline (for middle distillate sample like diesel, 200ml is taken) is distilled in a standard flask at a uniform rate of 5cc per minute.
2. The distillate is condensed in a brass tube condenser, surrounded by a water bath kept at 0^{o}C by ice –water mixture.
3. First drop from the condenser must be available in 5 to 10 minutes after heating is started , at which the recorded temperature is mentioned as initial boiling point (IBP) of the sample.
4. Heating is continued till 95% of the fraction is condensed. At this juncture , heat intensity may be increased to obtain the maximum boiling point also known as end point.
5. When the bottom of the flask shows dryness, the temperature recorded corresponds to final boiling point (FBP)

Observation:

1. Initial boiling point (IBP) of the sample =
2. Final boiling point (FBP) of the sample =

Result

1. Initial boiling point (IBP) of the sample =
2. Final boiling point (FBP) of the sample =

Questions:

1. List the test properties of gasoline.

2. Give the composition of gasoline.

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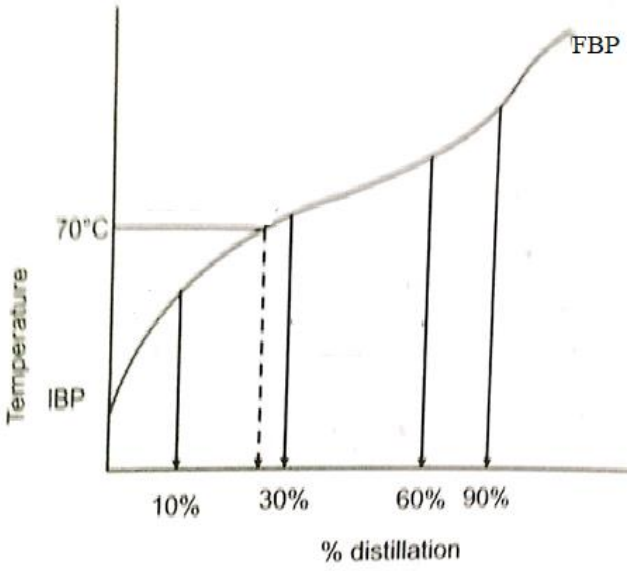
C(4)	P(4)	A(2)	TOTAL	SIGN

EXPERIMENT 12

Aim: Use the Draw distillation characteristic curve for given sample using ASTM distillation setup.

Theory: The most important characterization property of the crude streams is the ASTM distillation curve in which the boiling point of various volume fractions is measured at atmospheric pressure. The ASTM distillation is carried out in a single stage apparatus without any reflux.



ASTM distillation characteristic

Procedure:

1. 100 ml of gasoline (for middle distillate sample like diesel, 200ml is taken) is distilled in a standard flask at a uniform rate of 5cc per minute.
2. The distillate is condensed in a brass tube condenser, surrounded by a water bath kept at $0^{o}C$ by ice –water mixture.
3. First drop from the condenser must be available in 5 to 10 minutes after heating is started , at which the recorded temperature is mentioned as initial boiling point (IBP) of the sample.
4. The vapour temperature is recorded for each successive 10cc distillate collected in the measuring cylinder.
5. When the bottom of the flask shows dryness, the temperature recorded corresponds to final boiling point (FBP)
6. Plot a graph with temperature on Y axis and volume % of distillate collected on X axis

Observation:

Sample	Volume collected, ml	Volume %	Temperature

Result:

Distillation characteristic curve for the given sample is plotted.

Questions:

1. Define octane number and give its significance.
2. Define cetane number and give its significance.

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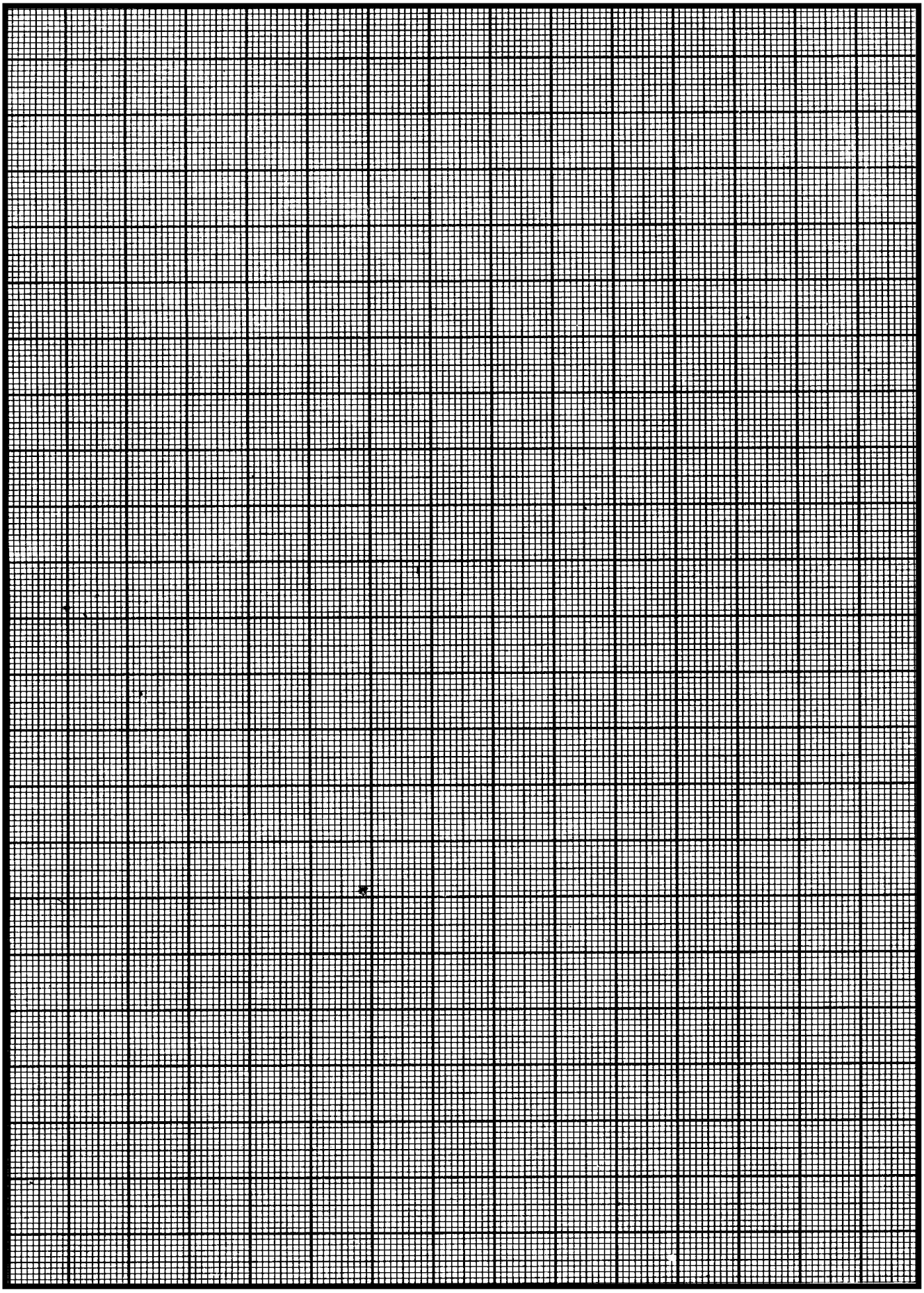
C(4)	P(4)	A(2)	TOTAL	SIGN



EXPERIMENT 13

Aim: Use the Esterification process to prepare Ethyl Acetate.

Safety Precautions:

Concentrated sulfuric acid can cause extreme burns if spilled on skin or in eyes. Use with caution and wear safety goggles at all times. Ether is extremely flammable and alcohols are somewhat flammable and they should be kept away from flames. Flask will be hot at end of refluxing, allow glassware to cool before removing. Stir with a spatula until CO_2 evolution is no longer vigorous.

Experimental set up:

Procedure:

1. Set up a reflux apparatus as shown in figure using a clean and dry condenser and an appropriate sized round bottom flask.
2. Measure 40 ml of 95 % of ethanol and add it to the round bottom flask.
3. Measure out 30 ml of glacial acetic acid, and add it to the round bottom flask already containing alcohol.
4. Add several boiling stones to the round bottom flask containing the alcohol and carboxylic acid.
5. Very slowly and carefully add 5ml of concentrated sulphuric acid, while swirling and cooling the flask.
6. Quickly reassemble the reflux apparatus, and heat the reaction for 45 min to 1 hour, while maintaining a steady reflux.
7. Remove the heating mantle and cool the reaction mixture to room temperature. You may speed the cooling up by placing the stoppered round bottom flask into a lukewarm water bath. Do not use an ice bath.
8. Pour the cooled mixture into a small separating funnel containing 20 ml of ice water. Rinse the round bottom flask with a further 5 ml of cold distilled water,

and add this also to the separating funnel .Put the stopper of the separating funnel and invert it several times.

9. Extract your ester with 25 ml of diethyl ether and separate the layers .Keep the aqueous layer. Do not discard anything yet.
10. Wash the crude ester (in the diethyl ether) with 25 ml cold distilled water [the purpose of this step is to wash away the water soluble impurities].
11. Wash the crude ester (in diethyl ether) with 25ml of 5% M sodium carbonate. Be extra careful to frequently vent the separating funnel, as you gently swirl the contents of the funnel. Do not invert the funnel at first. Carbon dioxide gas is formed during this step, and significant pressure builds up inside the funnel. When the amount of gas has declined, then invert and periodically vent the funnel.
12. Repeat the wash of the crude ester with another 25ml of 5 % sodium carbonate. Less CO_2 gas should be produced in this step than the previous.
13. Check the pH of the solution. It should be close to pH = 7.0.
14. Wash the crude ester with 25 ml of saturated sodium chloride. Withdraw the aqueous salt solution out the bottom of the funnel and pour the ester out the top of the separating funnel into a small clean , dry Erlenmeyer flask.
15. Dry the crude ester with anhydrous calcium chloride. Stopper and swirl the flask periodically for 15 min. be sure to add enough of the anhydrous drying agent so that some of it is still freely moving in the liquid. When the ester is dry, the crude ester should be clear and transparent; cloudiness indicates that water is still present.
16. Weigh the dry ester formed.

Results

The weight of ester formed is ----------

Questions:

1) What is esterification?

2) What is reaction involved in the esterification?

3) Which acid is used in the reaction?

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C(4)	P(4)	A(2)	TOTAL	SIGN

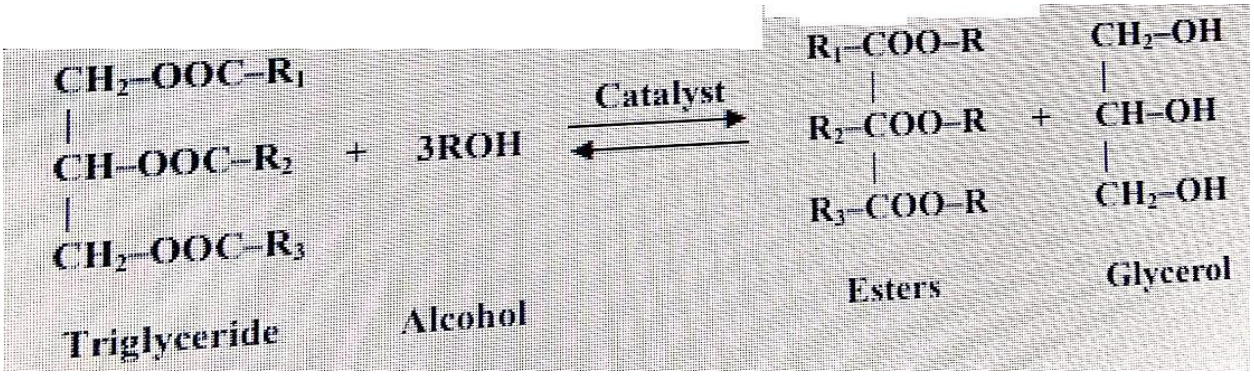
EXPERIMENT 14

Aim: Use the trans esterification process to prepare biodiesel from used oil.

Theory: Biodiesel is a diesel fuel that is made by reacting vegetable oil (cooking oil) with other common chemicals. Biodiesel may be used in any diesel automotive engine in its pure form or blended with petroleum-based diesel. No modifications are required, and the result is a less-expensive, renewable, clean-burning fuel. Trans esterification process is the most common way to produce biodiesel

Trans esterification is the process of exchanging the alkoxy group of an ester compound by another alcohol. In this process a glyceride reacts with an alcohol (typically methanol or ethanol) in the presence of a catalyst forming fatty acid alkyl esters and an alcohol. Vegetable oil molecule (triacyglycerols or triglycerides) comprises of 3 esters attached to a molecule of glycerine. When vegetable oil reacts with an alcohol (i.e., methanol), each triglyceride molecule is broken into three fatty acid chains or esters. The three esters are released from the glycerol skeleton and combine with the alcohol to yield fatty acid alkyl esters (i.e., fatty acid methyl esters or FAME). Sodium hydroxide (NaOH) is used as a catalyst to break the triglycerides. The catalyst then combines with glycerin and falls to the bottom of the container. Glycerol is produced as a by-product.

The reaction involved in the production of biodiesel and glycerol is



Equipment and apparatus required :

1. Rotamantle or magnetic stirrer
2. Helical Condenser
3. Separating funnel
4. Two mouthed conical flask
5. Thermometer
6. Stand for separating funnel and helical condenser
7. Retort ring for separating funnel and helical condenser
8. Rubber tubes for supply of water
9. Weighing balance

Reagents: 1. Methanol (CH3OH)

2. Sodium Hydroxide (NaOH)

Procedure:

a. The waste oil is filtered using a filter paper.
b. A certain amount of the filtered oil is taken in a conical flask and heated to 60^0C in the rotamantle to melt the lumpy particles in the oil. The magnetic needle in the rotamantle helps in uniform mixing and heating of oil.
c. Sodium hydroxide pellets are mixed with methanol and the solution is stirred continuously until the pellets are dissolved completely and a sodium methoxide solution is formed.
d. The sodium methoxide solution is poured slowly to the heated oil and the mixture is allowed to stir and heat for two hours at 60^0C. The helical condenser is fitted to the conical flask to prevent the methanol from vaporizing. A thermometer is inserted through the second mouth of the conical flask to keep a check on the temperature.
e. After stirring, the solution is poured into the separating funnel and is allowed to settle for 8 hours. A major part of the separation takes place in the first hour after the reaction and so a separation progress can be seen. Within 8 hours the glycerin will fall to the bottom of the separating funnel and the layer above glycerin is methyl esters which is lighter in colour than the bottom layer. The bottom layer comprises of glycerin, sodium hydroxide and methanol.
f. The stopcock at the bottom of the separating funnel drains the glycerin out in a container. When the glycerin is fully drained the valve is shut.
g. The glycerin left in the biodiesel sample comprises of soap which emulsifies when hot water is poured and forms a milky layer which is drained through the stopcock.

Result:

Questions.

1. Define trans esterification process.

2. Give the advantages of biodiesel.

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C(4)	P(4)	A(2)	TOTAL	SIGN

EXPERIMENT NO: - 16

Aim: Prepare Phenol formaldehyde resin using Phenol.
Apparatus: - Beaker, Conical flask, glass rod, measuring cylinder, fractional weight box etc.
Chemicals: - Glacial acetic acid, 40 % formaldehyde solution, phenol, conc. HCl, distilled water
Theory: - Phenol resins are condensation polymerization product of phenolic derivative with aldehyde (like formaldehyde, furfural) It is prepared by condensing phenol with formaldehyde in presence of acid or alkaline catalyst.
Reaction:-
Step I:- Formation of ortho and para hydroxy benzyl alcohols from phenol and formaldehyde:-

Step II:- Formation of Novalac from ortho-hydroxy,benzyl alcohol

Step III:- Formation of Bakelite from Novalac:-

Procedure:-
1) Place 5 ml of glacial acetic acid and 2.5 ml of formaldehyde solution in 500 ml beaker.
2) Add 2 grams of phenol and 1 ml of conc. HCl solution in it.
3) Heat the solution slowly with constant stirring for 5 minutes.
4) A large mass of pink plastic is formed.
5) The residue obtained is washed several times with distilled water.
6) Dry the product and calculate the yield accurately.

Result: - The weight of obtained Bakelite is …….. grams.

Questions:

1) What are thermoplastics?

2) What are thermosetting plastics?

3) Write the uses of plastics in day to day life.

4) Write the differences between thermosetting & thermoplastics.

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C(4)	P(4)	A(2)	TOTAL	SIGN