Energy and Environmental Science

Subject Code:

Experiment No. 4

Aim

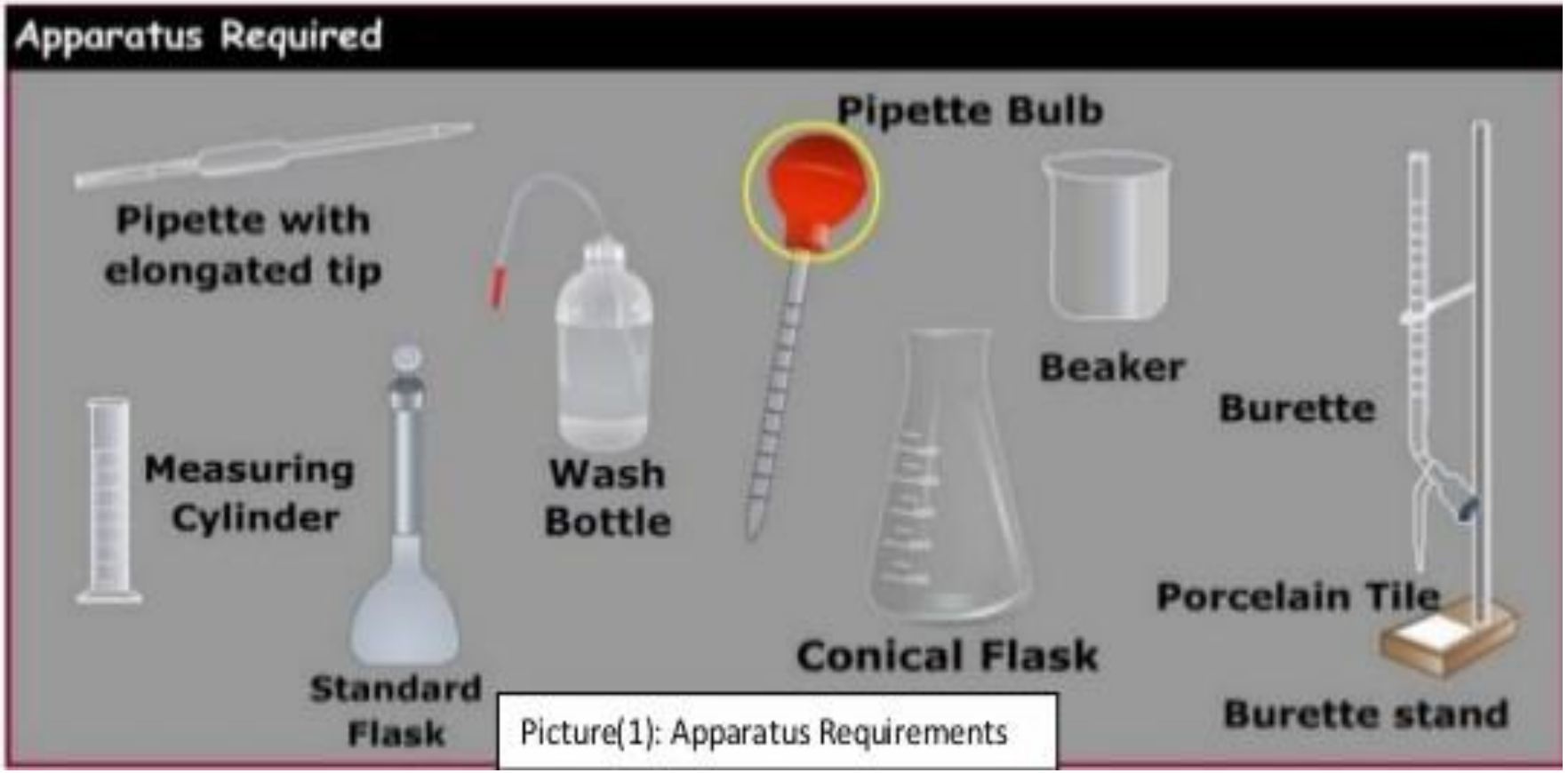
To determine the saponification value of given lubricating oil sample provided N/2 HCl alcoholic KOH solution.

Chemical requirement

- Phenolphthalein solution ,
- N/2 alcoholic KOH solution,
- N/2 HCl solution.

Apparatus requirement

- Iodine flask,
- burette,
- burette stand,
- air condenser, pipette etc.



Indicator used:

Phenolphthalein

End Point:

Pink to colorless

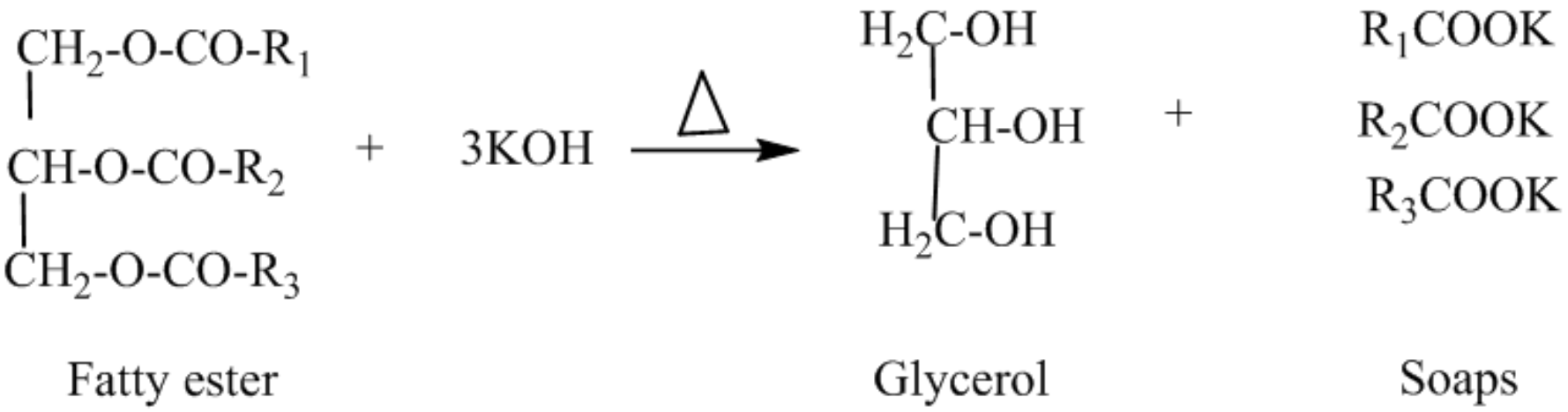
Theory

Saponification is the hydrolysis of an ester with base like NaOH or KOH solution to give alcohol and Na or K salt of the corresponding acid. Therefore, saponification value of an oil sample is the amount of KOH in mg required to saponify the fatty material present in 1g of lubricating oil. Mineral oils being a mixture of hydrocarbons do not react with KOH and hence are not saponificable, while vegetable and animal oils are mixture of glycerol esters or fatty esters and require large amount of alkali to get hydrolyzed.

For the determination of saponification value of given oil, a Known weight of oil sample is allowed to reflux with Known excess volume of standard KOH solution in a suitable solvent. Following chemical reaction takes place during the saponification of the oil sample.

Theory

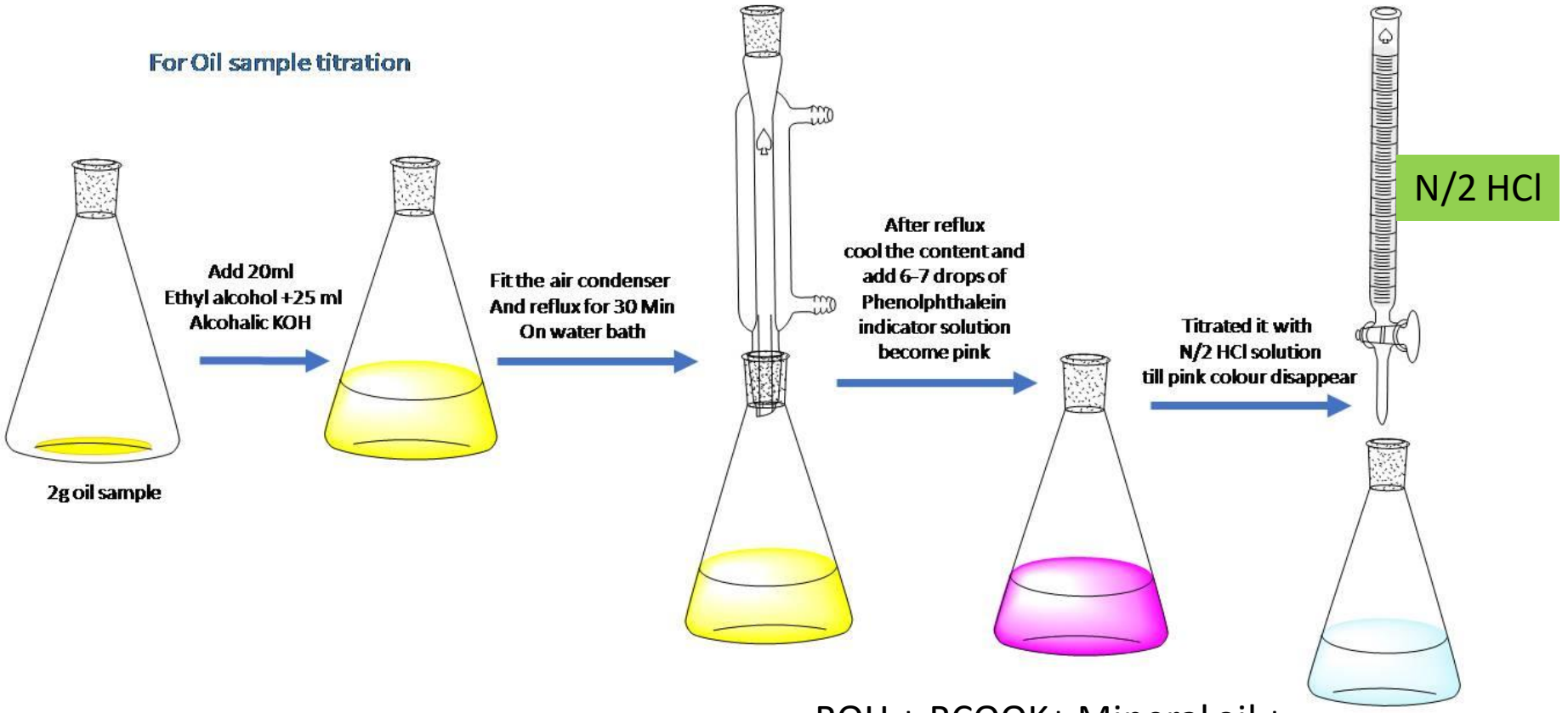
The un-reacted N/2KOH solution is then titrated with N/2HCl solution using phenolphthalein as an indicator. Also perform a blank experiment without oil sample.



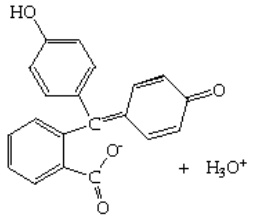
Procedure:

Take Known amount of oil sample (2g approx.) in a 250mL conical flask. Add 25mL of alcoholic KOH and 20mL ethanol. In another flask take same amount of alcoholic KOH and ethanol for the blank experiment. Fit the air condenser in both flasks and reflux the contents for half an hour. After 30 min. cool the flask and disconnect air condensers. Rinse the inner walls of the condensers with solvent into the flask and add few drops (8-10 drops) of phenolphthalein in both the flasks. Now, titrate the contents of these flasks against N/2 HCl until the pink color disappears and note the readings.

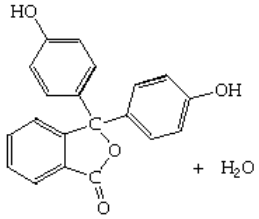
Procedure



ROH + RCOOK+ Mineral oil +
Unconsumed Alc KOH +
Coj. Base of Ph.

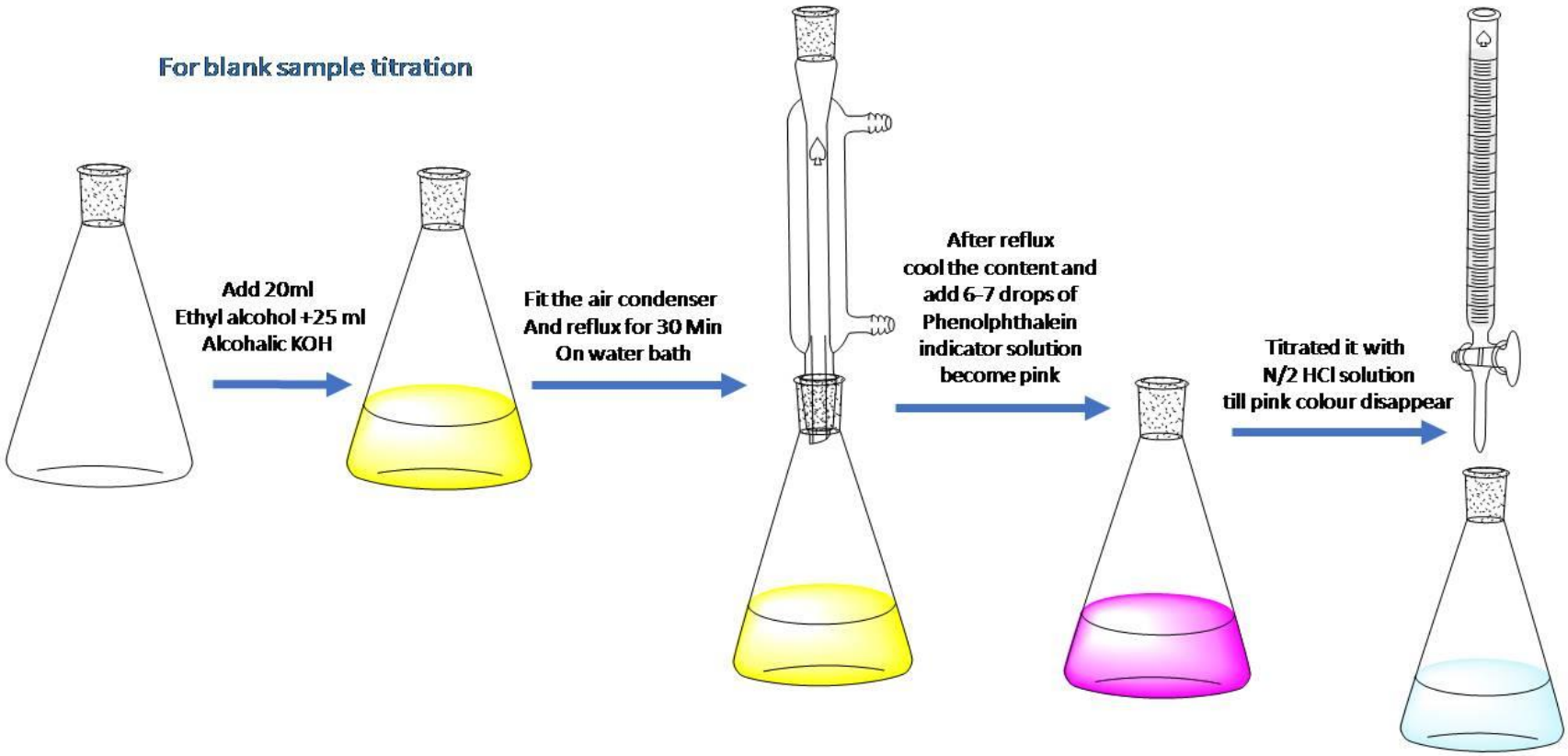


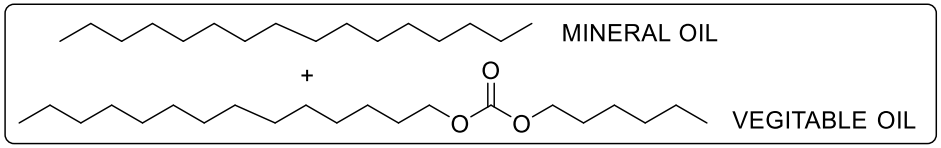
pink (Base)



colourless (Acid)

For blank sample titration



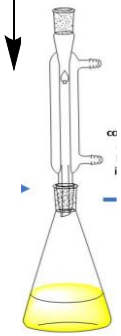


OIL SAMPLE

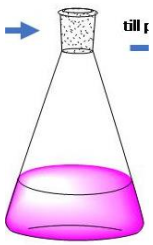
REFLUX

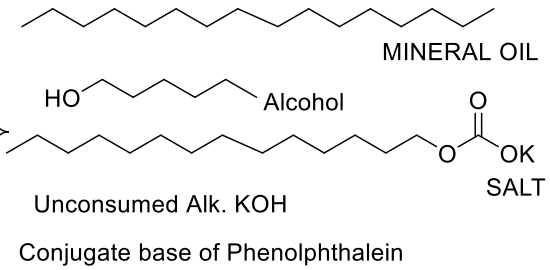
Dissolve in Ethyl alcohol

+ Alk. KOH



+ Phenolphthalein





Unconsumed Alk. KOH

Conjugate base of Phenolphthalein

TITRATION WITH N/2 HCl

Observations

Weight of the oil sample taken = W g
Titration of un-reacted N/2 KOH Vs N/2 HCI

S.No.	Contents	Burette reading Initial Final	Volume of N/2 HCl used (mL)
1	Sample		A
2	Blank		B

The volume of unreacted KOH solution left in each flask = Volume of HCl solution used for the titration of respective flask solutions (Because normality of KOH and HCl are same)

Calculation:

$$\text{Saponification value} = \frac{\text{Vol. of KOH consumed x Normality of KOH X Eq. wt of KOH}}{\text{Weight of oil}}$$

$$\text{Saponification value} = \frac{(B-A) \times 56}{2 \times W}$$

Results: Saponification value of oil sample = ……

Precaution

- The burette must be rinsed properly.
- HCl is colorless, so lower meniscus is noted as the correct reading.
- Parallax error must be avoided.
- Proper uniform refluxing should be done.

Significance:

- To distinguish between mineral oils (mixture of hydrocarbons) and fatty oils (esters). The saponification value of mineral oils is zero and high for vegitable oils.
- To identify the given fatty oil (specific saponification value for specific fatty oil).
- To determine the extent of adulteration.
- To determine the %of blending of the mineral oil.

Thank You