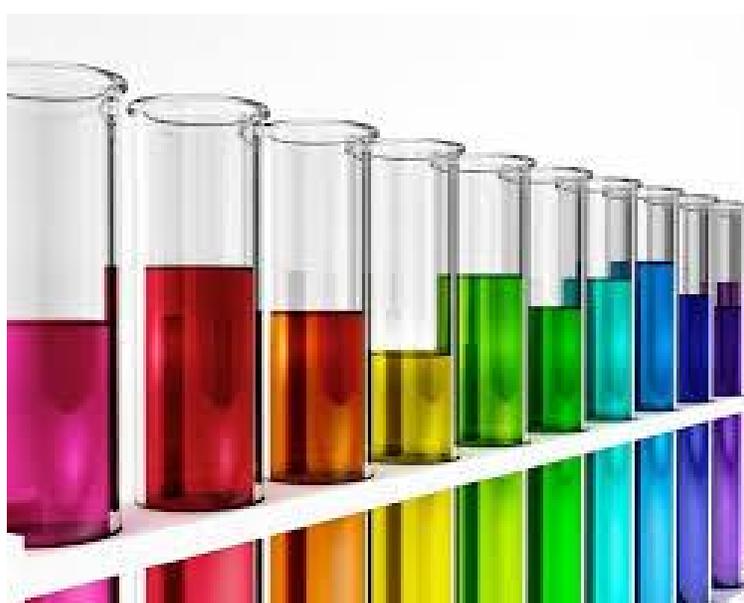


Shri Ramdeobaba College of Engineering and Management

**(An Autonomous Institute under Rashtrasant Tukadoji
Maharaj Nagpur University, Nagpur)**

Department of Applied Chemistry



Practical Manual

Session: 2015-2016

Shri Ramdeobaba

College of Engineering and Management

(An Autonomous Institute under Rashtrasant Tukadoji Maharaj Nagpur University,
Nagpur)

Department of Applied Chemistry

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EXPERIMENT NO. 1

Aim: To find out type of alkalinity and estimate alkalinity present in the given water sample.

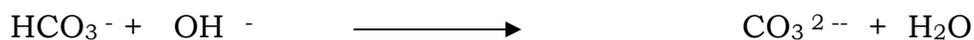
Chemicals: Std. Na_2CO_3 solution, Unknown water sample A and C, HCl solution (sample B) as an intermediate solution, phenolphthalein indicator and methyl orange/red indicator.

Principle:

The alkalinity of a water sample is due to carbonates (CO_3^{2-}), bicarbonates (HCO_3^-) and hydroxide (OH^-) ions. Thus in a given water sample the possible combination of ions causing alkalinity are as follows.

- (i) OH^- alone
- (ii) CO_3^{2-} alone
- (iii) HCO_3^- alone
- (iv) OH^- and CO_3^{2-} together
- (v) CO_3^{2-} and HCO_3^- together

The possibility of OH^- and HCO_3^- ions together in the same solution is ruled out as they react as-



In the same way, possibility of co-existence of all the three OH^- , CO_3^{2-} and HCO_3^- is ruled out.

The determination of alkalinity involves following reactions.





The phenolphthalein is pink in color above pH 10 and it is colorless below pH-8. While, the methyl orange is yellow above pH 4.4 and it turns pink below pH 3.1

Thus titration of a given water sample in presence of phenolphthalein as an indicator indicates completion of reaction 1 and 2 whereas the same water sample, if titrated in presence of Methyl orange as an indicator indicates the completion of reaction 1, 2 and 3.

The water sample, when titrated with an acid solution using phenolphthalein indicator gives (End point = P) and with methyl orange indicator gives (End point = M). The relation between P and M points gives the type and extent of alkalinity is established as follows:

Relation between P&M point	Type of alkalinity	Extent of alkalinity		
		OH ⁻	CO ₃ ²⁻	HCO ₃ ⁻
P = M	Only OH ⁻	M	-	-
P = 1/2 M	Only CO ₃ ⁻	-	2P	-
P = 0	Only HCO ₃ ⁻	-	-	M
P > 1/2 M	OH ⁻ & CO ₃ ⁻	(2P-M)	2(M-P)	-
P < 1/2 M	CO ₃ ⁻ & HCO ₃ ⁻	-	2P	(M-2P)

Procedure:

Part I: - Standardization of HCl solution using standard Na₂CO₃ solution.

Pipette out 25-ml std. Na₂CO₃ solution in a 250 ml conical flask. Add 1-2 drops of methyl orange/red indicator to it. The color of solution turns to yellow. Titrate this reaction mixture with HCl solution from the burette till the solution color turns to light pink/orange. This is the end point of titration. Repeat the same procedure to get successive constant end point.

Part II:- Estimation of type and extent of alkalinity present in sample A.

Pipette out 25-ml water sample A in a 250 ml conical flask. Add 1-2 drops of phenolphthalein as an indicator. The solution becomes pink. Titrate this solution with acid solution from the burette. At the end point the pink color of the solution changes to colorless. Note this end point as P point. Now At this point, add 2 drops of methyl orange indicator to the same solution. Solution becomes yellow. Continue the titration without refilling the burette the solution till the color of solution turns to orange / pink. Note this end point as M point of the titration. Repeat the same procedure to get successive constant readings of P and M point.

The relationship between P point and M point denotes the type and extent of alkalinity present in the given water samples as shown in earlier table.

Part III:- Estimation of type and extent of alkalinity present in sample C.

Pipette out 25-ml water sample C in a 250 ml conical flask and follow the same procedure as given in part II.

Observation table:

Part I: - Standardization of HCl solution using standard Na₂CO₃ solution.

Standard Na₂CO₃ solution Vs HCl solution

Control reading: to ml

S. No	Vol. of Na ₂ CO ₃ solution (V ₁ ml)	Volume of HCl solution (ml)		Constant Reading (V ₂ ml).
		IBR	FBR	

Part II:- Estimation of type and extent of alkalinity present in sample A.

Given water sample A Vs HCl solution

Control reading: P point: to ml

M Point: : to ml

S. No.	Vol. given water sample A (V_2 ml)	Volume of HCl solution (ml)			End Point (ml)
		IBR	P point	M point	
					P point= M point=

Part II:- Estimation of type and extent of alkalinity present in sample C.

Given water sample C Vs HCl solution

Control reading: P point: to ml

M Point: : to ml

S. No.	Vol. given water sample C (V_3 ml)	Volume of HCl solution (ml)			End Point (ml)
		IBR	P point	M point	
					P point= M point=

Calculations:-

Part I: - Standardization of HCl solution using standard Na₂CO₃ solution.

Given: Normality of Na₂CO₃ solution = N₁

Standard Na ₂ CO ₃ solution		HCl solution
$N_1 \times V_1$	=	$N_2 \times V_2$
		$N_1 \times V_1$
N_2	=	-----
		V_2

The given Normality of HCl solution is = N₂

Part II : Estimation of type and extent of alkalinity present in sample A

For sample A, P = ml and M = ml

If $P > 1 / 2 M$

then,

The type of alkalinity present in given water sample A is OH⁻ and CO₃²⁻

Now, 1) Volume of acid corresponding to OH⁻ = 2P- M

2) Volume of acid corresponding to CO₃²⁻ = 2 (M- P)

a) Normality of water sample due to OH-

HCl		Water sample A
$N_2 V_2$	=	$N_3 V_3$
		$N_2 \times 2P- M$
N_3	=	-----
		25

$$\begin{aligned}
 \text{Wt/ lt for OH}^- &= \text{Normality} \times \text{Eq. Wt.of CaCO}_3 \\
 &= N_3 \quad \times \quad 50 \\
 &= \quad \quad \quad \text{g/l} \\
 &= \quad \quad \quad \times 1000 \quad \text{mg/l} \\
 &= \quad \quad \quad \text{ppm}
 \end{aligned}$$

b) Normality of water sample due to CO₃²⁻.

HCl		Water sample A
N ₂ V ₂	=	N ₄ V ₄
	N ₂ X	2 (M- P)
N ₄	=	-----
		25

$$\begin{aligned}
 \text{Wt./ lt for CO}_3^{2-}. &= \text{Normality} \quad \times \text{Eq.. Wt. of CaCO}_3 \\
 &= N_4 \quad \times \quad 50 \text{ g/l} \\
 &= \quad \quad \quad \times 1000 \quad \text{mg/l} \\
 &= \quad \quad \quad \text{ppm}
 \end{aligned}$$

Same calculations has to be carried out for Water sample C

Result: -

The given water sample A contains:-

- OH⁻ Alkalinity = ----- ppm CaCO₃ equivalent.
- CO₃²⁻ Alkalinity = ----- ppm CaCO₃ equivalent.
- Total Alkalinity = ----- ppm CaCO₃ equivalent.

The given water sample C contains:-

- HCO₃⁻ Alkalinity = ----- ppm CaCO₃ equivalent.
- CO₃²⁻ Alkalinity = ----- ppm CaCO₃ equivalent.
- Total Alkalinity = ----- ppm CaCO₃ equivalent.

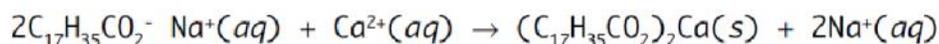
EXPERIMENT NO. 2

Aim : Estimation of temporary, permanent and total hardness present in supplied hard water sample by complexometric method.

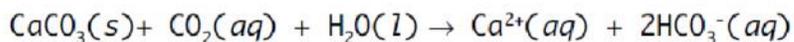
Chemicals Required: Standard hard water (sample A), given water sample (Sample C), ethylene diamine tetra-acetic acid solution as an Intermediate solution (Sample B), Buffer solution (NH₄Cl + NH₄OH having pH = 10), Eriochrome Black-T indicator solution.

Principle: Hard water contains the dissolved salts of calcium, magnesium and iron ions which are called hardening ions. In low concentrations these ions are not considered harmful for domestic use, but at higher concentrations of these ions interfere with the cleansing action of soaps and accelerate the corrosion of steel pipes, especially those carrying hot water.

Hardening ions, such as Ca²⁺ and Mg²⁺, form insoluble compounds with soaps. Soaps, which are sodium salts of fatty acids such as sodium stearate, C₁₇H₃₅CO₂Na, are very effective cleansing agents so long as they remain soluble; the presence of the hardening ions however causes the formation of a gray, insoluble soap scum such as (C₁₇H₃₅CO₂)₂Ca.

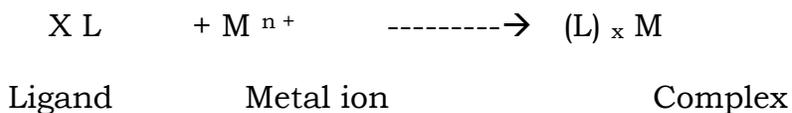


Groundwater becomes hard as it flows through underground limestone (CaCO₃) deposits; generally, the water from deep wells has a higher hardness than that from shallow wells because of a longer contact time with the limestone. Surface water similarly accumulates hardening ions as a result of flowing over limestone deposits. In either case the CO₂ dissolved in rainwater solubilizes limestone deposits.

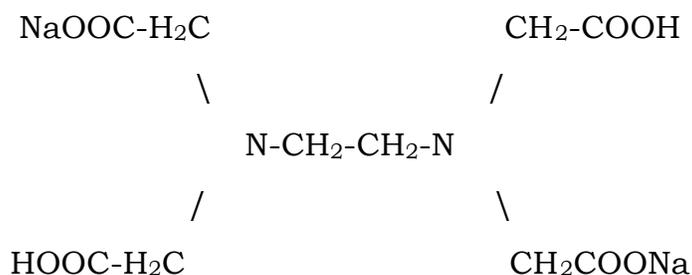


Hard water is also responsible for the appearance and undesirable formation of "boiler scale". The boiler scale is a poor conductor of heat and thus reduces the efficiency of transferring heat.

Complexometry involves the interaction of an organic compound with a complexable metal ion and results in the formation of compounds known as Werner's complexes. The organic compound is an anion or a Lewis base and is known as Ligand. Several factors, such as pH, basicity of ligand, type of ring formed etc. govern the successful formation of a complex. Thus:



In this experiment a titration technique is used to measure the combined Ca^{2+} and Mg^{2+} concentrations in a water sample. The titrant or the intermediate solution is the disodium salt of ethylene diamine tetraacetic acid (abbreviated $\text{Na}_2\text{H}_2\text{Y}$ or EDTA). Its structure is as below:



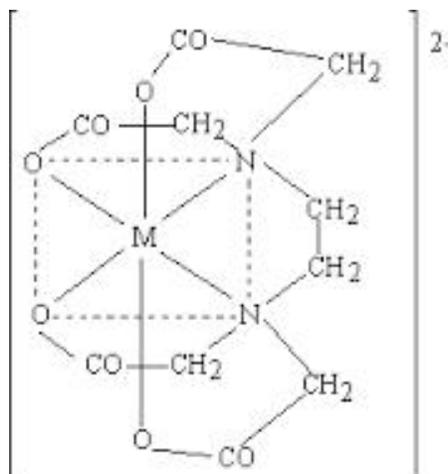
Disodium salt of EDTA

In aqueous solution $\text{Na}_2\text{H}_2\text{Y}$ dissociates into Na^+ and H_2Y^{2-} ions. The H_2Y^{2-} ion reacts with the hardening ions, Ca^{2+} and Mg^{2+} , to form very stable complex ions, especially in a solution buffered at a pH of about 10. As Ca^{2+} and Mg^{2+} is present in the hard water and EDTA solution are added from the burette, it complexes with the "free" Ca^{2+} and Mg^{2+} of the water sample to form the respective complex ions:



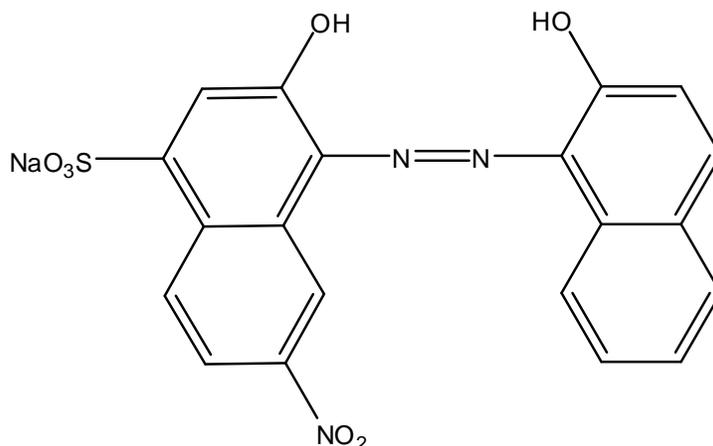
From the balanced equations, once the molar concentration of the $\text{Na}_2\text{H}_2\text{Y}$ solution is known, the moles of hardening ions in a water sample can be calculated from a 1:1 stoichiometric ratio. Thus it is evident that 1 mole of the complex forming H_2Y^{2-} reacts in with one mole of the metal ion and, two moles of hydrogen ions are generated. It is apparent from the equation above that the dissociation of the complex will be governed by the pH of the solution. Lowering of the pH will decrease the stability of the metal EDTA complex. Thus, pH should be maintained constant by the addition of basic buffer in the reaction mixture.

The structure of EDTA-metal complex is as follows:

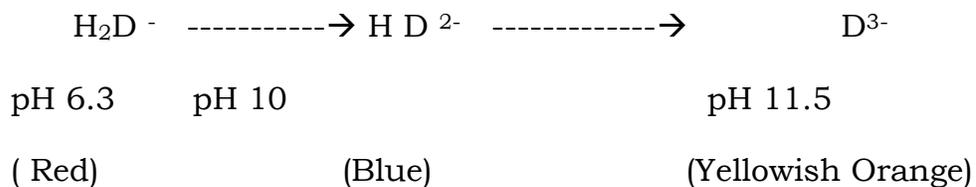


Calcium-EDTA Complex

An indicator is used to detect the endpoint in the titration, Eriochrome Black T (EBT). Eriochrome black-T is sodium 1 - (1-hydroxy, 2- naphthyl azo) 6-nitro - 2 naphthol-4, sulphonate.

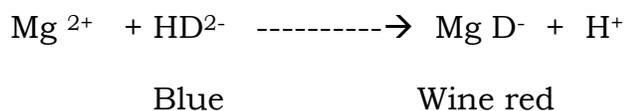


Hence EBT for the simplicity is represented as NaH_2D which gives H_2D^- ion which exhibits different colours at different pH values.



In the pH range 8-10, the blue form of the indicator HD^{2-} gives a wine red complex with Metal ion.

For ex.:



It forms complex ions with Ca^{2+} and Mg^{2+} ions, but binds more strongly to Mg^{2+} ions.

In this estimation, thus four types of complexes are possible

viz :

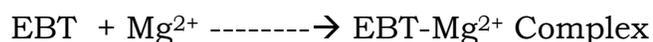
- a) EBT – Mg²⁺ complex (Wine red)
- b) EBT - Ca²⁺ complex (Wine red)
- c) EDTA- Mg²⁺ complex (colourless)
- d) EDTA- Ca²⁺ complex (colourless)

Their order of stability and consequently, their preference of formation are:



Because only a small amount of EBT is added, only a small quantity of Mg²⁺ is complexed; no Ca²⁺ ion complexes to EBT — therefore, most of the hardening ions remain "free" in solution. The EBT indicator is sky-blue in solution but forms a wine-red complex ion, [Mg-EBT]²⁺.

pH



8-10 (Wine red colour obtained)

Before any EDTA solution is added for the analysis, the reaction mixture is wine-red because of the [Mg-EBT]²⁺ complex ion. Once the EDTA complexes all of the "free" Ca²⁺ and Mg²⁺ from the water sample, it then removes the trace amount of Mg²⁺ from the wine-red [Mg-EBT]²⁺ complex ion. The solution changes from wine-red back to the sky-blue color of the EBT indicator, and the endpoint is reached — all of the hardening ions have been complexed with EDTA. The reaction is given as follows:

pH

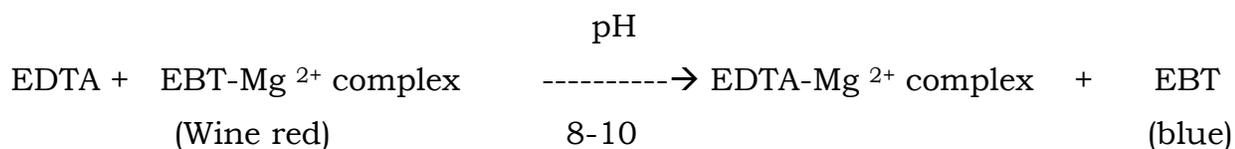


8-10

pH



8-10



PROCEDURE:

Part A: Standardization of EDTA solution using standard hard water sample

Pipette out 25 ml. of standard hard water solution in a clean conical flask. To it add 5 ml buffer solution (NH₄Cl + NH₄OH) and 3-4 drops of internal indicator EBT. The whole solution becomes wine-red in colour. Titrate this solution against EDTA solution from the burette. A change of colour of the solution from wine-red to blue indicates end point. Repeat the same procedure to get successive constant reading.

Part B: Estimation of total hardness present in given water sample C

Pipette out 25 ml. of the hard water sample C into a 250 ml. conical flask. To it add 5 ml buffer solution (NH₄Cl + NH₄OH) and 3-4 drops of internal indicator EBT. The whole solution becomes wine-red in colour. Titrate this solution against EDTA solution from the burette. A change of colour of the solution from wine-red to blue indicates end point. Repeat the same procedure to get successive constant reading.

Part C: Estimation of Parmanant hardness present in given water sample C

Pipette out 100 ml of given hard water sample C in 250 ml borosil conical flask. Boil this solution gently to reduce to 1/3rd of its original volume. Filter this warmed solution into 250 ml volumetric flask. After complete transfer of boiled sample, remove the filter paper. Wash the funnel properly with distilled water and collect the washings in the volumetric flask. Then make up the volume up to the mark of volumetric flask using distilled water. Shake the solution and then transfer it to 250 ml clean beaker.

Now pipette out 25 ml of diluted hard water sample C and add 5 ml buffer solution ($\text{NH}_4\text{Cl} + \text{NH}_4\text{OH}$) and 3-4 drops of internal indicator EBT to it. Titrate this reaction mixture with EDTA solution from the burette until the wine red colour changes into blue. Repeat the same procedure to get successive constant reading.

Observation Table:

Part 1: Standardization of EDTA solution using standard hard water sample

Standard Hard Water Sample Vs EDTA solution

Control reading: to ml

S. No.	Vol. of std. Hard water (V_1 ml)	Volume of EDTA solution (ml)		Constant Reading (V_2 ml).
		IBR	FBR	

Part 2: Estimation of Total Hardness present in given water sample C.

Water Sample C Vs EDTA solution

Control reading: to ml

S. No.	Vol. of given Hard water sample C (V_3 ml)	Volume of EDTA solution (ml)		Volume of EDTA solution
		IBR	IBR	

Part C: Estimation of Parmanant hardness present in given water sample C

Diluted Water Sample C Vs EDTA solution

Control reading: to ml

S.No	Vol. of Diluted Hard water sample C (V_3 ml)	Volume of EDTA solution (ml)		Volume of EDTA solution
		IBR	IBR	

Calculations:

For part 1: Standardization of EDTA solution using standard hard water sample

Let W g of hardness causing salt is dissolved in y ml of water.

$$W \times 1000$$

$$\text{Normality of std. Hard water} = \frac{\text{Eq. wt. of salt} \times y}{\text{-----}} = M_1$$

$$\begin{array}{ccc} \text{Std Hard water} & & \text{EDTA} \\ N_1 \times V_1 & = & N_2 \times V_2 \end{array}$$

$$N_2 = \frac{N_1 \times V_1}{V_2}$$

= (Normality of EDTA)

Part 2 : Determination of Total Hardness present in given water sample.

$$\begin{array}{ccc} \text{EDTA} & & \text{Water Sample} \\ N_2 \times V_2' & = & N_3 \times V_3 \\ & & N_2 \times V_2' \\ N_3 & = & \text{-----} \\ & & V_3 \\ & & = \text{(Normality of hard water)} \end{array}$$

Therefore the amount of Total Hardness present in given water sample can be calculated as:

$$\begin{aligned} \text{Wt/Lit} &= N_3 \times \text{Equivalent Wt. of CaCO}_3 \times 1000 \\ &= \mathbf{A} \quad \text{mg/ lit CaCO}_3 \text{ equivalent hardness.} \end{aligned}$$

Part C: Estimation of Parmanant hardness present in given water sample C

$$\begin{array}{ccc} \text{Diluted Water Sample C} & & \text{EDTA solution} \\ N_2 \times V_2'' & = & N_3 \times V_3 \\ & & N_2 \times V_2'' \end{array}$$

$$N_3 = \frac{\text{-----}}{V_3}$$

= (Normality of diluted hard water)

Therefore the amount of Permanent Hardness present in diluted water sample can be calculated as:

$$\begin{aligned} \text{Wt/Lit} &= N_3 \times \text{Equivalent Wt. of CaCO}_3 \times 1000 \\ &= \mathbf{B} \text{ mg/ lit CaCO}_3 \text{ equivalent hardness.} \end{aligned}$$

This much amount of hardness is present in diluted water sample C.

But, 250 ml diluted water sample C is prepared from 100 ml water sample C i.e. 2.5 times diluted.

Hence, permanent hardness in water sample C = B X 2.5 mg/ lit CaCO₃ equivalent hardness.

Therefore, Temporary hardness present in given water sample C

$$= (A-B) \text{ mg/ lit CaCO}_3 \text{ equivalent hardness}$$

Result: The amount of hardness present in given sample C is as follows:

Permanent Hardness = mg/ lit CaCO₃ equivalent hardness

Temporary Hardness = mg/ lit CaCO₃ equivalent hardness

Total Hardness = mg/ lit CaCO₃ equivalent hardness

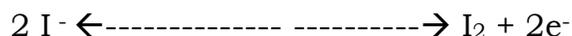
EXPERIMENT NO. 3

AIM: Estimation of copper present in an alloy by iodometry.

THEORY: -

The Iodometric method of volumetric analysis is based on oxidation -reduction process (Redox) and involves interconversion of elemental Iodine into Iodide ions and vice-versa as-

Iodometry



Iodimetry

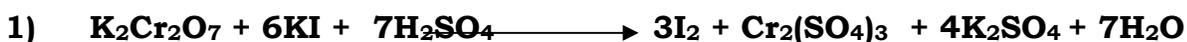
There are two types of Iodometric titrations

- Indirect Iodometric titrations (**Iodometry**) : It involves titration of liberated iodine from potassium iodide (KI) in a chemical reaction.
- Direct Iodometric titrations (**Iodimetry**) : It involves titration with Standard solution of Iodine.

PRINCIPLE:-

In this Experiment, an oxidizing agent such as $K_2Cr_2O_7$ or $CuSO_4$ is treated in acid solution (dil. H_2SO_4) with an excess of iodide ion produced by KI. The oxidizing agent oxidizes the iodide ions into Iodine, which is then titrated with the standardised solution of reducing agent such as sodium thiosulphate solution. (Hypo)

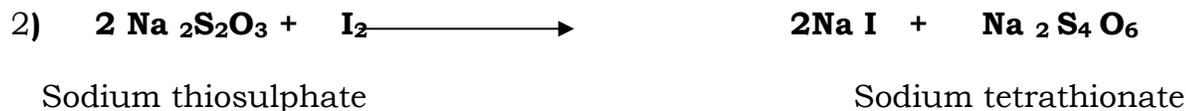
PART A: Standardization of Sodium thiosulphate solution.



In ionic form, the same reaction can be written as



Thus the amount of Iodine liberated depends only upon the quantity of $\text{K}_2\text{Cr}_2\text{O}_7$ act as limiting reagent.



In ionic form, the same reaction can be written as



PART B: Estimation of copper ions:



In ionic form, the same reaction can be written as



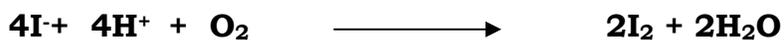
The amount of iodine thus generated depends upon the concentration of Cu^{2+} ions which can be estimated by titrating it against sodium thiosulphate solution using starch as an indicator.

Since the solubility of Iodine in water is low, a considerable excess of KI must be used in iodometric determination of oxidizing agent. The iodine liberated by the reaction then dissolves by forming the complex $\text{K}(\text{I}_3)$



Formation of this compound $(\text{I}_3)^-$ does not interfere with in the titration of iodine with Sodium thiosulphate because, as the free iodine reacts with thiosulphate, the equilibrium between I_2 and $(\text{I}_3)^-$ is disturbed and more I_2 is produced by the backward reaction.

After addition of KI and /or acid the flask is kept in dark place for about 5 minutes as rate of the reaction between oxidizing agent and I^- is too low.. Secondly, light accelerates side reaction of photo-oxidation as,



Here, I⁻ are oxidized to I₂ by atmospheric oxygen and analytical results may go wrong. It is for this purpose, before adding KI to the acidic solution of the oxidizing agent, a pinch of Na₂CO₃ is added so as to generate a CO₂ atmosphere which would prevent access of the O₂ to the solution.

Indicator:

Most commonly used indicator in iodometric titration is **STARCH**. It forms deep blue complex with iodine (but not with iodide). Starch is a polymer, consisting of two components Amylose and Amylopectine. The active fraction amylose is a polymer of the sugar α -D- glucose. It has a shape of coiled helix into which I₂ combined with I⁻ can fit, thus reacting slowly with thiosulphate. Due to this reason the starch solution must be added at the very end of the titration (when iodine conc. is very less).

Starch solution is not very stable solution because various microorganisms are degrading it and one of the degradation products, glucose, is a reducing agent. Thus an old starch solution containing some glucose can cause an appreciable titration error. Hence always fresh starch solution is used.

PROCEDURE: -

Part A: -Standardization of sodium thiosulphate solution.

Pipette out 25ml. std. K₂Cr₂O₇ solution in a conical flask. Add 10 ml.dil. sulphuric acid, followed by a pinch of sodium bicarbonate /sodium carbonate and finally 10 ml KI (10%). Keep the flask covered for 5-6 minutes in dark. Appearance of dark brown color indicates the formation of I₂.

Now, titrate it against Na₂S₂O₃ from burette until dark brown colour becomes faint brown. At this stage, add 2 ml. starch solution, colour of the solution becomes

intense blue. Continue titration drop wise till blue color disappears. Note this point as an end point.

Part B: -Estimation of Cu²⁺ ions.

Pipette out 25 ml. given CuSO₄ solution in a conical flask. Add few drops of dil. Na₂CO₃ solution until a faint permanent precipitate remains. Add acetic acid solution dropwise till the solution becomes clear. Now add 10 ml. KI (10%). Appearance of dark brown colour indicates formation of I₂. Titrate the liberated iodine with Na₂S₂O₃ solution from burette till brown colour of iodine fades, then add 2ml. of starch solution, and continue the addition of the Na₂S₂O₃ solution until the blue color commences to fade. Then add about 5-6ml of ammonium thiocyanate solution into the flask due to which blue color become more intense. Continue the titration till blue color disappears (there is formation of flesh coloured ppt in the flask). Note this point as an end point.

Calculations:-

Given Normality of std. K₂Cr₂O₇ is N₁

Part A: -Standardization of Sodium thiosulphate solution.(Hypo.)

Std. K ₂ Cr ₂ O ₇	v/s		Hypo
N ₁ V ₁	=		N ₂ V ₂
N ₁ x 25	=		N ₂ x V ₂
			N ₁ x 25
N ₂	=		-----
			V ₂
N ₂	=	X	(Normality of hypo solution)

Part B: -Estimation of Cu²⁺ ions.

Hypo v/s given CuSO₄

$$N_2V_2 = N_3V_3$$

$$N_3 = \frac{N_2V_2}{25}$$

$$N_3 = Y \quad (\text{Normality of CuSO}_4 \text{ solution.})$$

$$\begin{aligned} \text{Wt./lt. of Cu}^{2+} &= \text{Normality} \times \text{Eq. Wt. Of Cu}^{2+} \text{ ions} \\ &= N_3 \times 63.54 \\ &= Z \text{ g/l} \end{aligned}$$

Result: Hence, The given solution of copper salt was found to contain Z g/l of Cu²⁺ ions.

Precautions:

- 1) To suppress the volatility of Iodine, excess of KI is added.
- 2) Flask is to be kept in dark to avoid photo oxidation of Iodide ions to Iodine.
- 3) Freshly prepared starch solution only can be used as an indicator.

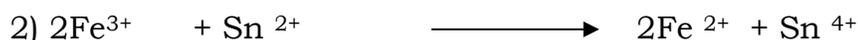
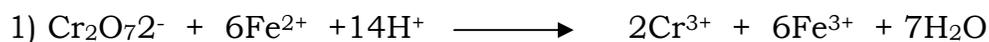
EXPERIMENT NO. 4

Aim: - Estimation of Ferrous, Ferric and total iron content present in an ore by Redox titrimetry.

Principle:

The principle involved in this titration is that only Ferrous ions gets oxidized to ferric ions and the ferric ions in the solution remains as it is.

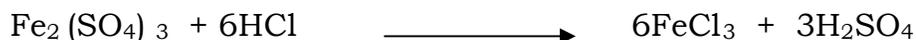
The amount of ferrous ions present in the given solution containing ferrous and ferric salt is determined by titrating the solution, and then the total iron (present now in the ferrous state only) is titrated with potassium dichromate.



In acid solution reduction of potassium dichromate may be represented as:



Ferric salts are not oxidized either by dichromate or permanganate. They can be determined in solution only after reduction of Fe^{3+} to Fe^{2+} ions. This reduction can be carried out by reducing agents such as SnCl_2 . Ferric salt e.g. FeCl_3 is reduced by the action of SnCl_2 in presence of hydrochloric acid.



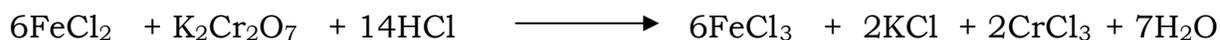
The excess SnCl_2 must be removed completely, as $\text{K}_2\text{Cr}_2\text{O}_7$ oxidizes it. This is done by adding mercuric chloride, which reacts with SnCl_2 as follows:



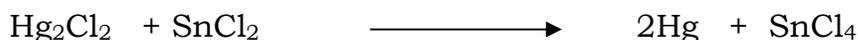
(Excess added)

(Silky white ppt.)

Mecurous chloride (calomel Hg_2Cl_2) is deposited in the form of silky white precipitate. This is not filtered off and the solution is directly titrated with $\text{K}_2\text{Cr}_2\text{O}_7$, Following reaction takes place: -



The following points must be taken into consideration in this analysis. The precipitated Hg_2Cl_2 can also oxidize by dichromate. However, if only a small excess SnCl_2 is used in the previous stage, the amount of HgCl_2 precipitated is small, it reacts with the dichromate slowly and no appreciable error is introduced into the result. On the other hand, if a large excess of Hg_2Cl_2 is precipitated and, in particular, if it has a gray or dark colour as a result of further reduction to metallic mercury by the reaction



Then it is oxidized so rapidly in the titration that the result is quite erroneous.

It follows, therefore, that a basic condition for accuracy in this method is the use of a very slight excess of SnCl_2 . Stannous chloride is added drop by drop until the reddish yellow/ yellow colour of FeCl_3 has completely disappeared, after which one or two more drops are added.

PROCEDURE:

Part-I : Estimation of Ferrous ions present in given mixture:

Pipette out 25 ml. of the given Ferrous & Ferric salt solution mixture in a conical flask, add about 10 ml. of sulphuric acid and 5-6 drops of N-Phenyl anthranilic acid as an indicator. Titrate slowly with constant shaking of the flask against $\text{K}_2\text{Cr}_2\text{O}_7$ solution till the brick red colour, which marks the end point. This titre value corresponds to the ferrous ions present in the solution.

Part-II : Determination of total iron content present in given mixture.

Pipette out 25 ml. of the given Ferrous & Ferric salt solution mixture in a conical flask and add 10 ml. of conc. HCl, The solution turns reddish yellow due to the formation of ferric chloride. Heat the flask and to the hot solution (70-90°C) add concentrated stannous chloride solution (SnCl₂) drop wise with constant stirring till the solution becomes colourless. Add one drop extra to ensure complete reduction.

Cool the solution rapidly under the tap water. Add 5 ml of Mercuric chloride at once. A silky white precipitate of mercurous chloride is formed. [However, if a heavy precipitate forms, or a gray or black precipitate is obtained, it means that too much of SnCl₂ has been used. If no precipitate was formed, it shows that SnCl₂ added was insufficient. In either of these above cases, the expt. has to be rejected and fresh start should be made.]

To this white ppt containing solution, add 4-5 drops of N-Phenylantranilic acid as an indicator. Titrate it against std. K₂Cr₂O₇ solution, till the solution turns brick red. Mark this point as end point.

CALCULATIONS:

Part-I : Estimation of Ferrous ions present in given mixture:

$$\begin{aligned}
 &\text{Std. K}_2\text{Cr}_2\text{O}_7 \quad \text{vs} \quad \text{Fe}^{2+} / \text{Fe}^{3+} \text{ mixture} \\
 &N_1V_1 = N_2V_2 \\
 &N_2 = \frac{N_1V_1}{V_2} \\
 &N_2 = \text{----- (Normality of Solution w.r.to Fe}^{2+}\text{)} \\
 &\text{Wt./ lit.of Fe}^{2+} = N_2 \times 55.85 \\
 &= X \quad \text{g/l}
 \end{aligned}$$

Part-II : Determination of total iron content present in given mixture.

$$\begin{aligned}
 &\text{Std. K}_2\text{Cr}_2\text{O}_7 \quad \text{vs} \quad \text{Fe}^{2+} / \text{Fe}^{3+} \text{ mixture} \\
 &N_1V_1 = N_3V_3 \\
 &N_3 = \frac{N_1V_1}{V_3} \\
 &N_3 = \text{----- g/l (Normality w.r.to Total iron content)} \\
 &\text{Wt./ lit.of} = N_3 \times 55.85 \\
 &= Y \text{ g/l}
 \end{aligned}$$

So, The amount of Fe^{3+} ions present in the mixture = $(Y - X)$ g/l .

Result: The amount of Fe^{2+} , Fe^{3+} and total iron content present in given mixture was found to be _____ respectively.

EXPERIMENT NO. 5

Aim: Determination of relative and kinematic viscosities of a given lubricating oil at different temperatures using Redwood Viscometer No. 1 or No. 2.

Apparatus: Redwood viscometer (1 / 2), thermometer, etc.

THEORY AND GENERAL DISCUSSION:

Viscosity is one of the most important properties of a lubricating oil. Viscosity is a measure of the internal resistance to the motion of a fluid and is mainly due to the forces of cohesion between the fluid molecules. The formation of a fluid film of a lubricant between the moving friction surface and the generation of the frictional heat under the particular condition of load, bearing speed and lubricant supply is mainly depends upon the viscosity of the lubricant and to some extent on its oiliness. If the viscosity of the oil is too low, the fluid lubricant film cannot be maintained between the moving surfaces as a result of which excessive wear may take place. On the other hand, if the viscosity of the oil is too high, excessive friction due to the shearing of oil itself would result. Hence in hydrodynamic lubrication, the lubricant should possess the proper viscosity. So, it is of vital importance to have knowledge of the viscosity of the lubricating oil. Viscosity of the fluid may be measured in several ways, one of which is determining time required for a definite amount of the liquid to flow through a capillary. Such method includes the use of Saybolt, Engler and Redwood Viscometers.

Viscosity may be expressed as dynamic (or absolute) viscosity, kinematic viscosity or the viscosity called after the name of the apparatus used for its determination.

DYNAMIC OR ABSOLUTE VISCOSITY:

Dynamic or absolute viscosity (often denoted by 'n') is the tangential force per unit area required to maintain unit velocity gradient between two parallel planes, in the

fluid unit distance apart. It can be also be defined as the ratio of shearing stress to the rate of shearing strain.

If F is the force required to keep moving an particle of surface area A in contact with the fluid, separated from stationary surface by a thickness D, and moving with velocity V, then

$$\begin{aligned} \text{Shearing stress} &= \text{Force acting on the surface area A} \\ \text{i.e. } F/A &: \text{-----(1)} \end{aligned}$$

$$\text{Rate of shearing strain} = V/D \text{-----(2)}$$

Absolute Viscosity η = Shearing stress/ rate of shearing strain.

$$\frac{F / A}{V / D} = \frac{F \times D}{V \times A} \text{-----(3)}$$

The numerical value of ‘ η ’ depends upon the unit used in equation (3). In metric system the unit is ‘poise’ (F = 1 dyne, A = 1 cm. And V = km/sec.). A smaller unit, the centipoises is more often used. Poise is equal in one dyne/second/cm² (Dimensions of absolute viscosity are ML⁻¹T⁻¹). Absolute is also referred to as ‘coefficient of viscosity’.

KINEMATIC VISCOSITY: -

The ratio of absolute viscosity to density for any fluid is known as absolute kinematics viscosity. It is donated by μ and in C.G.S. system, its units are stokes and centistokes (1/100th of stoke) respectively.

$$\mu = \eta / \rho$$

Where,

$$\mu = \text{Absolute kinematic viscosity}$$

η = absolute dynamic viscosity

ρ = density of fluid.

Dimension of μ are $L^2 T^{-1}$

Since the rate, at which a fluid will flow through an apparatus increases as the internal friction of the fluid decreases, the rate of flow through an orifice or short tube may be used as a means for measuring viscosity. This is the principle involved in the Redwood viscometer, which is an English standard whereas Saybolt's Viscometer is used in U.S.A. and Engler's Viscometer in Europe. In these commercial Viscometers a fixed volume of the liquid is allowed to flow (in case of Redwood it is 50 ml. at 270C Engler-60 ml. and Saybolt -200 ml.) through a capillary tube of specified dimension under given set of conditions and the time of flow is measured at a particular temperature. The result is usually expressed in terms of the time in seconds taken by oil to flow through the standard orifice of the particular standard apparatus used.

e.g.: Viscosity of oil is 250, Redwood (no. ½) seconds at 270C. This Viscosity so determined is sometimes called as **Relative Viscosity**.

Absolute and kinematic Viscosities can also be determined from the relative Viscosity (i.e. Redwood values) from the equations:

$$\mu = Ct \text{ -----(5) (for Fluids)}$$

Whose Kinematic Viscosity is more than 10 centistokes

$$\text{and } m = Ct - \beta / t \text{ -----(6) (for fluids Kinematics Viscosities less than or Equal to 10 centistokes)}$$

Where,

μ = kinematic viscosity in centistokes

t = time of flow in seconds

C = viscometer constant

ρ = coefficient of kinetic energy which may be determined experimentally or eliminated by choosing long flow-times

Test Viscometer may be calibrated constant C determined by solutions of known Viscosity. The primary standard used is freshly distilled water, whose kinematics viscosity is 1.0008 centistokes. Other standards usually employed are-

40% sucrose solution

μ = 4.390 cs at 25 0C p = 1.1739

60% sucrose solution.

μ = 33.66 cs. At 250C p = 1.28335

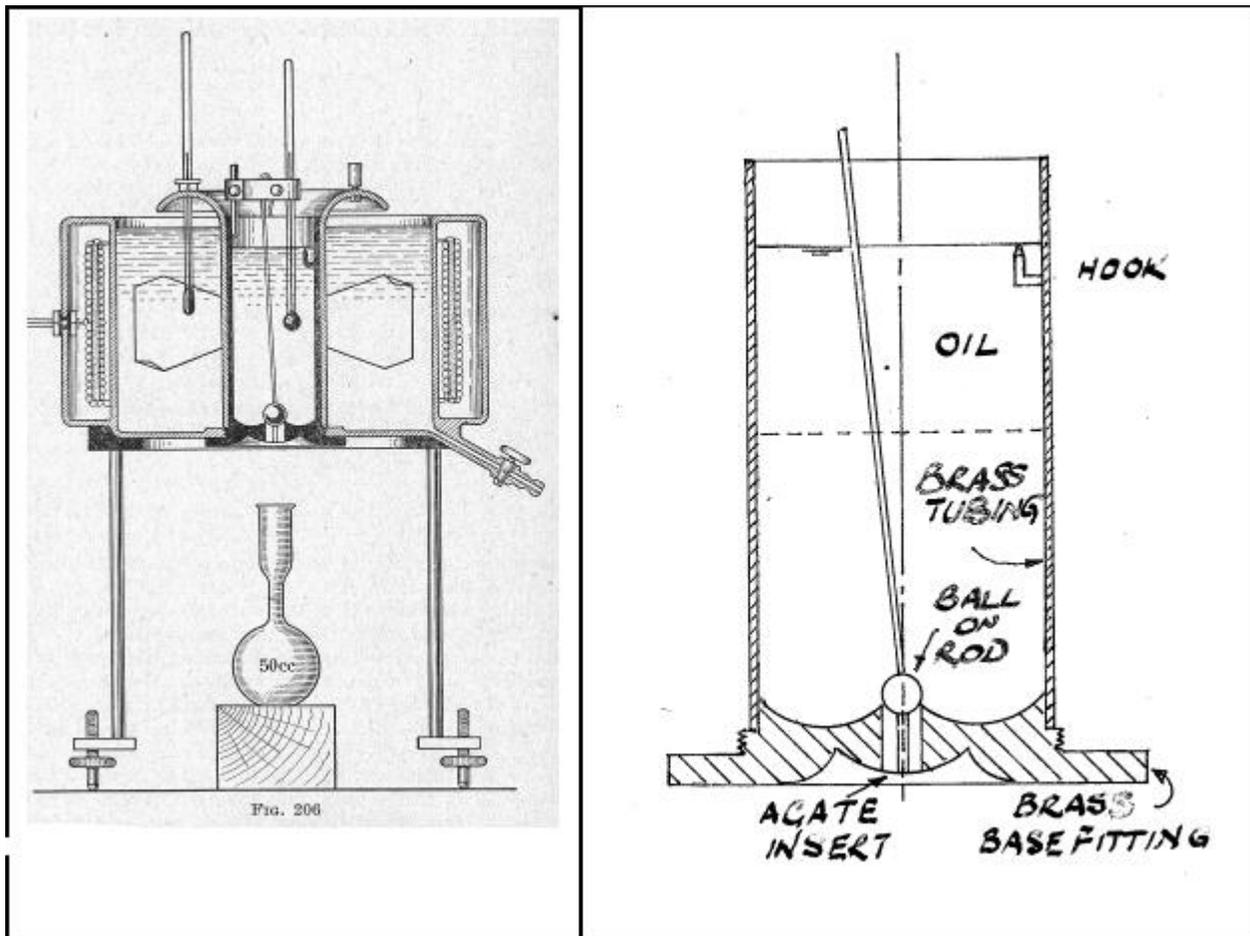
For Redwood viscometer No.1, the values for the constants are as below:

Time of flow, t	β	C
40 to 85 seconds	190	0.264
85 to 2000 seconds	65	0.247

For Redwood viscometer No.2, the values for the constants are **β =1120, C= 2.720**

Redwood viscometer No. 2 is used for very viscous liquids and gives 1/10 th the value of Redwood No.1 viscometer. Replace the ball value in position to seal the cap to prevent overflow of the oil.

Diagram:



PROCEDURE:

- 1) Level the instrument by leveling screws ensuring that it is horizontal with the help of spirit level.
- 2) Put the valve rod at its position (i.e. in the concavity at the bottom of the cup) to close the passage of orifice.
- 3) Keep clean dry Kohlrausch's flask centrally below the jet.
- 4) The test sample is poured into the oil cup and adjust the level of oil, with that of the pointer. Later indicates the level to which oil is to be filled.

- 5) Fill up the water bath with water and adjust the level of water with that of the pointer in the oil cup.
- 6) Cover the oil cup with the lid and insert thermocouple / thermometer of desired range into the oil cup from the thermometer bracket. Care should be taken that it does not touch the bottom of the cup.
- 7) Also insert the thermometer into the water bath from the shield.
- 8) Let the oil in the cup as well as water of the water bath attain the room temperature. Once the temperature of oil and water bath become steady for a period of 2-3 minutes record the temperatures separately. (Quite likely that there may be a difference of 1-2°C between the temperature of oil and the temperature of water in the water bath.
- 9) Lift up the valve rod and suspend it from the thermometer bracket and start the stop watch simultaneously.
- 10) Receiving flask is so located that oil strikes the flared mouth and does not drop directly into the opening, which would cause foaming.
- 11) When the level of the oil reaches 50 ml. mark in the neck of the flask, stop the stop-watch.
- 12) At the same time close the passage of the orifice by keeping the valve rod at its original position to prevent any overflow of oil.
- 13) The time elapsed in seconds is the relative viscosity of oil at room temperature.
- 14) Switch on the water bath adjust the knob of the regulator in such a way that the temperature of the water bath is a few degree above temperature i.e. 85°C
- 15) Again, pour oil into the cup and adjust oil level as described earlier.
- 16) Stir the contents of the bath and dup regularly.

- 17) When the temperature of sample has become quite steady at the desired value i.e. at 85°C lift the valve rod and suspend it from the thermometer bracket and start the stopwatch simultaneously.
- 18) When the level of the oil touches the 50 ml. mark on the neck of the flask stop the watch. The time elapsed in seconds in the relative viscosity of oil at 85°C.
- 19) Now take out some hot water from the water bath and add equal amount of cold water to bring the temperature of the bath slightly above that of test temperature i.e. 75°C.
- 20) Pour oil into the cup and adjust oil level as described earlier.
- 21) Stir the contents of the bath and cup regularly.
- 22) When the temperature of sample has become quite steady at the desired temperature i.e. 75°C find out the relative viscosity of test sample at this temperature as described earlier.
- 23) Repeat the procedure to find out relative viscosities of oil at 65°C, 55°C, 45°C, respectively.
- 24) Record all the results in the tabular form.
- 25) Construct graph co-relation (a) Viscosity and temperature, (b) Log of viscosity and temperature and (c) Kinematic viscosity and temperature (d) Log of Kinematic viscosity and temperature, (e) Find rate of change of relative viscosity w. r. t. Temperature of the given oil.

Observation table: Redwood viscometer no.:

No.	Sr.	Temperature (T) in °C	Time in second	Relative Viscosity (y_r)	Kinematic Viscosity (\sim) in centistroke	Log (y_r)	Log (\sim)
1		Room Temp.:					
2		85					
3		75					
4		65					
5		55					
6		45					
7		35					
8							

Calculation:

Graphs were plotted between

1. Relative viscosity (y_r) against the temperature of sample (T)
2. Log of relative viscosity (y_r) against the temperature of sample (T)
3. Kinematic viscosity (\sim) against the temperature of sample (T)
4. Log of kinematic viscosity (\sim) against the temperature of sample (T)

And determine the slope of the graph 1 from the plot and calculate it using the least square method.

Result:- The rate of change of relative viscosity with respect to temperature was found to be equal to

EXPERIMENT NO. 6

Aim: Determination of flash point of a combustible liquid by Able's closed cup flash point apparatus.

Theory:-

The Flash Point of oil may be defined as the minimum temperature to which it must be heated to give off sufficient vapour to ignite momentarily or less than 5 seconds when a flame of standard dimensions (Approx. 4 mm) is brought near the surface of the sample for a prescribed rate in an apparatus of specified dimensions. This is detected by the appearance of momentary flash upon the application of small flame over the surface of oil. The Flash Point is defined as closed cup or open cup flash point accordingly as the apparatus for the determination of flash point of sample is provided with a cover to cover the sample cup or not.

Whereas the Fire Point of oil may be defined as the minimum temperature to which it must be heated to give off sufficient vapour to ignite for more than 5 seconds when a flame of standard dimensions (Approx. 4 mm) is brought near the surface of the sample for a prescribed rate in an apparatus of specified dimensions.

The mechanism of the appearance of the flash can be explained in the following manner. Every flammable liquid has a vapour pressure, which is a function of the liquid's temperature. As the temperature increase, the vapour pressure increases, as the vapour pressure increases, the concentration of evaporated flammable liquid in the air increases. Hence, temperature determines the concentration of its vapour in the air to sustain combustion. The flash point of a flammable liquid is the lowest temperature at which there can be enough flammable vapour to ignite, when an ignition source is applied. Oil containing minute quantities of volatile organic substances is liable to flash below the true flash point of the oil. Although a small flash may be observed in such cases, it should not be confused with the true flash

point, since its intensity does not increase with increase in temperature, as occurred when the true flash point is reached.

Importance of flash point from view of lubricants:

Good lubricating oil should not volatilize under the working temperatures. Even if some volatilization takes place, the vapour formed should not form inflammable mixture with air under the condition of lubrication. From this point of view, the flash point of lubricating oil is of vital importance.

Lubricating oil selected for the job should have a flash point which is reasonably above its working temperature. This insures the safety against the fire hazards during the storage, transport and use of the lubricating oil. This test is immense importance for illuminating and lubricating Oils. This helps in detecting the highly volatile constituents of the oils. If they are highly volatile at ordinary temperature, the issuing vapors may cause fire hazards. So to ensure safety, certain minimum temperatures are laid down for fuels and Lubricating Oils below which they should not give off adequate vapors to make them burn.

Outline of the method:

The sample is placed in the oil cup of apparatus and heated at a slow uniform rate about 2 °C. A small test is directed into the cup at regular intervals, and the lowest temperature at which application of the test flame causes the vapor to ignite momentarily, with a distinct flash inside the cup, is recorded as Abel's Flash Point.

Description of the apparatus:

The **Abel's** apparatus consists of the following essential parts: -

SAMPLE CUP: This is a cylindrical vessel with a lid. Within the cup, near the top; there is a sample level mark.

COVER: The cup is provided with a close fitting cover. The cover is provided with a thermometer socket, to support an oil test lamp, a movable metal bead. The top of the cover has three rectangular holes which are covered or uncovered by moving the

slide which has two perforations to allow air to enter the oil up and bring contact between vapors of the oils and flame of the test-lamp when in open position.

A metal bead, the dimension (4mm) of which represents the size of test flame, is mounted in the cover. The apparatus is also provided with a stirrer.

HEATING VESSEL: -

The heating vessel consists of copper vessels and placed coaxially, one inside the other. The space between the two vessels is used as a water jacket. When the oil cup is placed into the hole at the top of the vessel, it fits into it and leaves an air gap between itself and outer copper water vessel.

The water can be heated electrically or with a burner or spirit lamp. Thermometer is provided with the apparatus for the measuring the temperature of the oil sample.

PROCEDURE:

Fill the given sample in such a way that the sample level is exactly up to the mark in the cup. Fix the cup in to the apparatus and cover with lid. Insert thermometer in the thermometer holder given in the cup in such a manner that it will not directly touch the lower bottom of the cup and the paddle stirrer inside the cup. Fill the water bath with the cold water. Close the sliding shutter and light the standard flame. Adjust the size of flame (4mm diameter) with respect to the metal bead. Stir the oil using paddle stirrer. Introduce the flame by opening the shutter and check the appearance of the flash. Now heat the apparatus and set the rate of temperature increase at the rate of 1 to 2 °C per minute. Check the flash point of given sample at the interval of 3 °C rise in the temperature. Discontinue the stirring the sample during the introduction of the test flame. On observing a flash, stop the heating process and allow the temperature to decrease. Check the occurrence of a flash at every 1 °C drop in temperature at which the flash is observed as the flash point of the sample.

Diagram:



OBSERVATION: Sample No.:-

Table 1

S. No.	Increasing Temperature (°C)	Inference (No flash or Flash observed)
1		
2		
3		
4		
5		
6		

Table 2

S. No.	Decreasing Temperature (°C)	Inference (No flash or Flash observed)
1		
2		
3		
4		
5		
6		

Result: The Flash Point of given Sample oil No. _____ by **Able'** Flash Point apparatus is found to be -----°C.

EXPERIMENT NO. 7

Aim: - Determination of flash point of a combustible liquid by Pensky-Martian's closed cup flash point apparatus.

Theory:-

The Flash Point of oil may be defined as the minimum temperature to which it must be heated to give off sufficient vapour to ignite momentarily or less than 5 seconds when a flame of standard dimensions (Approx. 4 mm) is brought near the surface of the sample for a prescribed rate in an apparatus of specified dimensions. This is detected by the appearance of momentary flash upon the application of small flame over the surface of oil. The Flash Point is defined as closed cup or open cup flash point accordingly as the apparatus for the determination of flash point of sample is provided with a cover to cover the sample cup or not.

Whereas the Fire Point of oil may be defined as the minimum temperature to which it must be heated to give off sufficient vapour to ignite for more than 5 seconds when a flame of standard dimensions (Approx. 4 mm) is brought near the surface of the sample for a prescribed rate in an apparatus of specified dimensions.

The mechanism of the appearance of the flash can be explained in the following manner. Every flammable liquid has a vapour pressure, which is a function of the liquid's temperature. As the temperature increase, the vapour pressure increases, as the vapour pressure increases, the concentration of evaporated flammable liquid in the air increases. Hence, temperature determines the concentration of its vapour in the air to sustain combustion. The flash point of a flammable liquid is the lowest temperature at which there can be enough flammable vapour to ignite, when an ignition source is applied. Oil containing minute quantities of volatile organic substances is liable to flash below the true flash point of the oil. Although a small

flash may be observed in such cases, it should not be confused with the true flash point, since its intensity does not increase with increase in temperature, as occurred when the true flash point is reached.

Importance of flash point from view of lubricants:

Good lubricating oil should not volatilize under the working temperatures. Even if some volatilization takes place, the vapour formed should not form inflammable mixture with air under the condition of lubrication. From this point of view, the flash point of lubricating oil is of vital importance.

Lubricating oil selected for the job should have a flash point which is reasonably above its working temperature. This insures the safety against the fire hazards during the storage, transport and use of the lubricating oil. This test is immense importance for illuminating and lubricating Oils. This helps in detecting the highly volatile constituents of the oils. If they are highly volatile at ordinary temperature, the issuing vapors may cause fire hazards. So to ensure safety, certain minimum temperatures are laid down for fuels and Lubricating Oils below which they should not give off adequate vapors to make them burn.

DESCRIPTION OF THE APPARATUS

A Pensky Martin apparatus consists of the following parts:

SAMPLE CUP: Sample cup is a cylindrical vessel, made of brass with a filling mark grooved inside near the top. It is provided with a lid.

LID: The lid is equipped with the following parts:

Stirrer: The stirring device consists of a vertical steel shaft mounted in the center of the cup and carrying two-bladed brass propellers.

Cover: It has four opening, one for thermometer and the rest for the oxygen entry and exposure of vapors to test flame.

Shutter: The lid is equipped with a brass shutter operating on the plane of the upper surface of the cover. The shutter is so shaped and mounted on the lid that when in one position, the holes are completely closed and when in the other, these orifices are completely opened.

The flame exposure device: The lid is equipped with a pilot lamp with such a mechanism that its flame operates simultaneously with the shutter. When the shutter is in the 'open' position, the tip is lowered down in the center of the central orifice.

Heater: The cup is heated by means of burner or it is electrically heated. The air bath has cylindrical interior about 4 cm. deep and can be heated by a direct flame or an electric resistance element. The top-plate is also made of metal and mounted with an air gap between it and the air bath.

Digram:



PROCEDURE:

The cup and its accessories are well cleaned and dried before the test is started. Now the cup is filled with the oil to be tested up to the level indicated by the filling mark and covered with the lid. The stirring device, the thermometer and flame exposure device is fixed on the top of the cover. The cup is now set in the apparatus properly and the thermometer inserted. The test flame is lighted and adjusted until it is the size of a bead (4mm in diameter). The apparatus is heated so that the heating rate is maintained, with the help of a rheostat, at 5-6 °C per minute and stirring rate at 1 to 2 rps.

Once the heating started, the test flames is applied after each 2 0C rise of temperature nearer to the sample in the cup by opening the shutter and check the appearance of the flash.

On observing a flash, stop the heating process and allow the temperature to decrease. Check the occurrence of a flash at every 1 °C drop in temperature. Record the lowest temperature at which the flash is observed as the flash point of the sample.

OBSERVATION:

Table 1

S. No.	Increasing Temperature (°C)	Inference (No flash or Flash observed)
1		
2		
3		
4		
5		
6		

Table 2

S. No.	Decreasing Temperature (°C)	Inference (No flash or Flash observed)
1		
2		
3		
4		
5		
6		

RESULT: The Flash Point of given sample determined by **Pensky Marten's Flash Point** apparatus is found to be -----°C.

EXPERIMENT NO. 8

Aim: Determination of flash point of a combustible liquid by Cleveland's open cup flash point apparatus.

DEFINITIONS:

Theory:-

The Flash Point of oil may be defined as the minimum temperature to which it must be heated to give off sufficient vapour to ignite momentarily or less than 5 seconds when a flame of standard dimensions (Approx. 4 mm) is brought near the surface of the sample for a prescribed rate in an apparatus of specified dimensions. This is detected by the appearance of momentary flash upon the application of small flame over the surface of oil. The Flash Point is defined as closed cup or open cup flash point accordingly as the apparatus for the determination of flash point of sample is provided with a cover to cover the sample cup or not.

Whereas the Fire Point of oil may be defined as the minimum temperature to which it must be heated to give off sufficient vapour to ignite for more than 5 seconds when a flame of standard dimensions (Approx. 4 mm) is brought near the surface of the sample for a prescribed rate in an apparatus of specified dimensions.

The mechanism of the appearance of the flash can be explained in the following manner. Every flammable liquid has a vapour pressure, which is a function of the liquid's temperature. As the temperature increase, the vapour pressure increases, as the vapour pressure increases, the concentration of evaporated flammable liquid in the air increases. Hence, temperature determines the concentration of its vapour in the air to sustain combustion. The flash point of a flammable liquid is the lowest temperature at which there can be enough flammable vapour to ignite, when an ignition source is applied. Oil containing minute quantities of volatile organic substances is liable to flash below the true flash point of the oil. Although a small flash may be observed in such cases, it should not be confused with the true flash

point, since its intensity does not increase with increase in temperature, as occurred when the true flash point is reached.

Importance of flash point from view of lubricants:

Good lubricating oil should not volatilize under the working temperatures. Even if some volatilization takes place, the vapour formed should not form inflammable mixture with air under the condition of lubrication. From this point of view, the flash point of lubricating oil is of vital importance.

Lubricating oil selected for the job should have a flash point which is reasonably above its working temperature. This insures the safety against the fire hazards during the storage, transport and use of the lubricating oil. This test is immense importance for illuminating and lubricating Oils. This helps in detecting the highly volatile constituents of the oils. If they are highly volatile at ordinary temperature, the issuing vapors may cause fire hazards. So to ensure safety, certain minimum temperatures are laid down for fuels and Lubricating Oils below which they should not give off adequate vapors to make them burn.

DESCRIPTION OF APPARATUS:

The apparatus consists of a test cup made of without any lid and is equipped with a handle. The cup is supported by a metal plate known as heating plate. The cup may be heated by an electric heater mounted below the cup in the apparatus itself

The metal plate has an extension for mounting the test flame and the thermometer support. The test flame is mounted in such a manner as to permit automatic duplication of the sweep of the test flame over the sample cup. The size of the flame can be adjusted with respect to the dimension of metal bead (4 mm).

Digram:



PROCEDURE:

The apparatus is thoroughly cleaned and the thermometer is suspended in such a way so that the bottom of the thermometer bulb just above the bottom of sample cup.

The cup is now filled with sample up to the filling mark grooved on the inner side of the cup taking care that the surface of the sample is free from bubbles and there is no oil above the filling mark. The test flame is adjusted to have a flame diameter of about 4 mm. Now move the test flame over the sample cup and check the appearance of flash over the sample inside the cup.

If no flash observed, increase the temperature of the sample taken and take a flame over the cup after every 2 °C increases. On observing a flash, stop the heating

process and allow the temperature to decrease. Check the occurrence of a flash at every 1 °C drop in temperature. Record the lowest temperature at which the flash is observed as the flash point of the sample.

OBSERVATION TABLE:

Table 1

S. No.	Increasing Temperature (°C)	Inference (No flash or Flash observed)
1		
2		
3		
4		
5		
6		

Table 2

S. No.	Decreasing Temperature (°C)	Inference (No flash or Flash observed)
1		
2		
3		
4		
5		
6		

Result:

The Flash Point of given sample determined by Cleveland's apparatus is found to be ----^oC.