Ajit Singhal '201500052'

EXPERIMENT - 1

Object:

To prepare and standardize the solution of NaOH (N/10) against standard solution of Oxalic acid (N/5)

Apparatus/Chemicals used:

Burette, Pipette, conical flask, Measuring cylinder, NaOH Solution, Phenolphthalein indicator (Internal Indicator), Oxalic acid (N/5)

Theory:

Standard solution is one in which exact amount of a substance is present in a definite volume of the solution, or a solution whose concentration (strength) is known to us is also called as standard solution.

Volumetric solutions are classified into following two types:

(i)Primary Standard Solution

(ii)Secondary Standard solution

Primary Standard Solution: The substance whose standard solution is prepared by dissolving directly its known amount in a definite volume of solvent or solution is known as a primary standard substances & the solution is called as primary standard solution. Commonly used primary standard substances are anhydrous Na_2CO_3, Oxalic acid etc.

Secondary Standard Solution: The substance whose solution cannot be prepared directly by weighing its definite amount and then dissolving in definite volume of solvent is called secondary standard substance & the solution is called as secondary standard solution. The solution of this type of substance firstly prepared is of approximate strength which is then standardized with a standard solution of a primary standard substance. The common secondary standard substances are alkali hydroxides, inorganic acids and $KMnO_4$ etc.

Classification of Methods of Volumetric Analysis:

Volumetric analyses are of following types

(i)Neutralization titrations or Acid Base titrations

(ii)Oxidation-reduction titrations

(iii)Precipitation titrations

(iv) Complexometric titrations

Normality (N):

The normality of a solution is the number of gram-equivalents of the solute per litre of the solution.

N = No. of grams equivalent of solute/ Volume of the solution in 1000mL

$(COOH)_2.2H_2O + 2NaOH \rightarrow Na_2C_2O_4 + 4H_2O$

Observations:

S/No.	NaoH Solution taken for titration (ml)	Initial readings	Final readings	Volume of standardized solution of oxalic acid consumed (ml)
1.	10 ml	0.0	9.7	9.7 ml
2.	10 ml	9.7	19.4	9.7 ml
3.	10 ml	19.4	29	9.6 ml

Calculations:

From Normality Equation:-

$N1V1 = N2V2$

(unknown solution NaOH) = (known solution oxalic acid)

$N1 \times 10 = N/5 \times 9.7$

$N1 = 0.194$

Calculation for dilution:-

$0.194 \times V1 = 0.1 \times 100$

$V1 = 51.54$ ml

Result:

Standard solution of N/10 NaOH solution is prepared.

Precautions:

1. Solution should be making up to desired volume after complete the solute.
2. During titration the solution should be stirred thoroughly
3. Do not take mean of burett readings.

EXPERIMENT - 2

Object:

To determine the constituent and amount of alkalinity in the given water sample by titrating it against standard HCl solution (N/20) using phenolphthalein and methyl orange as an internal indicators.

Materials/Chemicals/Apparatus required:

N/20 HCl solution, phenolphthalein and methyl orange indicators, sample solution, burette, pipette, conical flask, beaker, funnel, etc.

Theory:

The alkalinity in water is due to the presence of hydroxyl ion ($\mathbf{OH^-}$), carbonate ion ($\boldsymbol{CO3^{2^-}}$) and bicarbonate ion ($\boldsymbol{HCO3^-}$) present in the given sample of water. These can be estimated separately by titrating against standard acid (N/20 HCl) using phenolphthalein and methyl orange as indicators. The chemical reaction involved can be shown by the equations given below:

(i) $\boldsymbol{OH^- + H^+ \rightarrow H2O}$

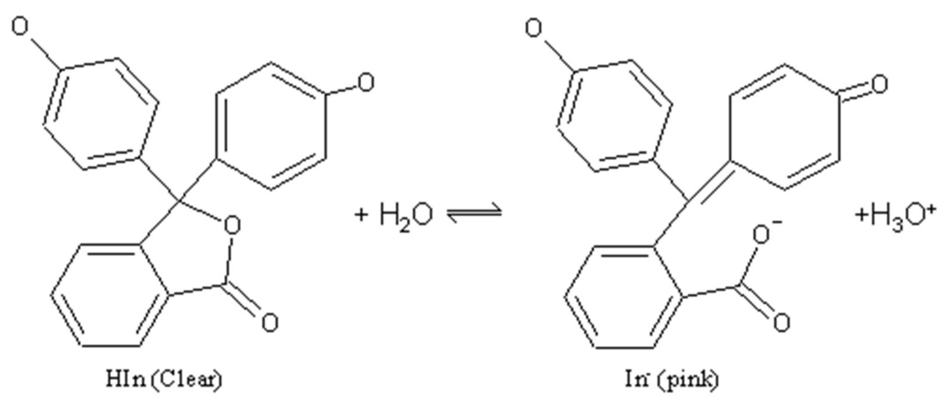
(ii) $\boldsymbol{CO3^{2^-} + H^+ \rightarrow HCO3^-}$

(iii) $\boldsymbol{HCO3^- + H^+ \rightarrow H2O + CO2}$

[P]-Stands for phenolphthalein indicator and [M]-stands for methyl orange indicator. In solution OH and HCO3 ions can't co-exist due to following reaction

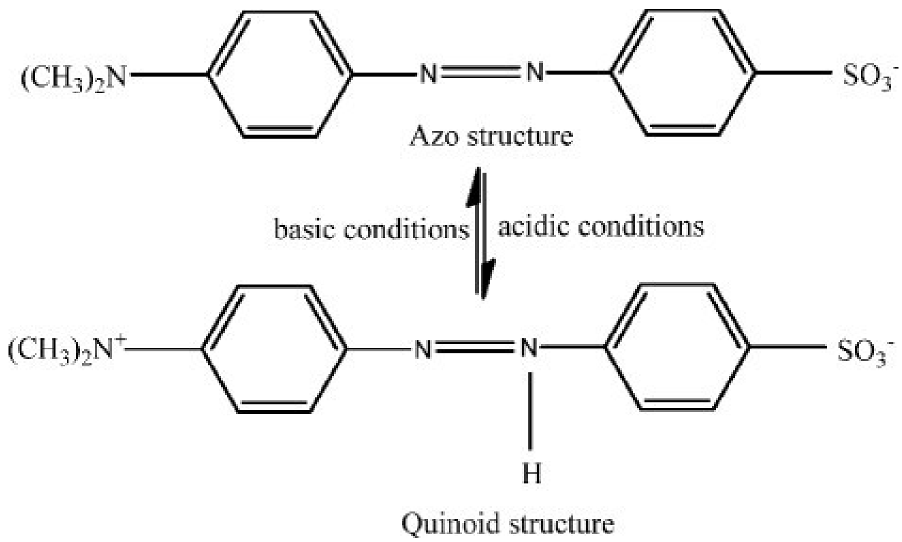
$$\boldsymbol{OH^- + HCO3^- \rightarrow H2O + CO3^{2^-}}$$

Phenolphthalein Structure:



+

Methyl orange Structure:



Observations:

S/No.	P Initial	P Final	M Initial	M Final	Volume of HCL with phenolphthalein (p) in ml	Volume of HCL with methyl orange (M) in ml
1.	0	0.6	0.6	2.5	0.6	1.9
2.	2.5	3.1	3.1	5	0.6	1.9
3.	5	5.5	5.5	7.4	0.5	1.9

Calculations:

Burette reading of phenolphthalein (p)=0.6 ml

Burette reading of phenolphthalein (m)=1.6 ml

As p<m = $CO3^{+} + HCO3^{-}$ *are present*

For $CO3^{-}$*:*

H20 HCL

N1V1 = N2 V2

N1 x 10 = 1/20 x 2p

N1 =1/200 x 2 x 0.6

N1=0.006

Strength= N1 x Equivalent weight of $CO3^{-}$

= 0.006 x 50gm/l

= 300 ppm

For $HCO3^{-}$*:*

$HCO3^{-}$ *HCL*

N1V1 = N2 V2

N1 x 10 = 1/20 x (m-p)

N1 =1/200 x 1.3

N1=0.0065

Strength= N1 x Equivalent weight of $HCO3^{-}$

= 0.0065 x 50gm/l

= 325 ppm

Result:

Strength of $CO3^{+}$*: 300ppm*

Strength of $HCO3^{-}$*: 325ppm*

Precautions:

1. Phenolphthalein indicator should be added first and then methyl orange.

2. The volume of indicator should same in all the titrations.

3. The reaction mixture should be shaken

4.*Do not take mean of burette readings*

EXPERIMENT - 3

Object:

To determine chloride ion content in a given water sample by Argentometric method (Mohr's method) using K_2CrO4 as an internal indicator.

Material/Chemicals/Apparatus required:

Burette, pipette, conical flask, measuring flask, standard silver nitrate solution (N/40), potassium chromate (K_2CrO4), etc.

Theory:

Chloride ions are present in water usually as NaCI, MgCl2 and CaCl2. Although chloride ions are not harmful as such but their concentration over 250 ppm imparts a peculiar taste to the water thus rendering the water unacceptable for drinking purposes.

By Argentometric method chloride ions in a water sample, which is neutral or slightly alkaline, can be determined by titrating it against standard silver nitrate solution using potassium chromate as an internal indicator.

$$Ag^{+} + \ Cl^{-} \ \rightarrow \ AgCl\text{(white ppt)}$$

$$2Ag^{+} + \ CrO4^{2^{-}} \ \rightarrow \ Ag2CrO4\text{(Brick red ppt)}$$

Brick red colour formed due to formation of silver chromate disappears as the solution contains high concentration of Cl^{-}

$$Ag_2CrO4 + 2\ Cl^{-} \rightarrow 2AgCl + CrO4^{2^{-}}$$

When the concentration of chloride ions has decreased, the red colour starts disappearing slowly on shaking and a stage is reached when all the chloride ions have formed AgCl ppt., then one extra drop of AgNO3 at this point reacts with potassium chromate and forms a reddish coloured ppt of silver chromate.

$$2AgNO3 + K_2CrO4 \rightarrow AgaCrO4 + 2KNO3$$

Observation:

(A)

S/No.	**Volume of distilled water (ml) (V1)**	**Burette Initial readings**	**Burette Final readings**	**Volume of used AgNO3 soln.(ml)(V)**
1.	10 ml	0.0	0.5	0.5ml
2.	10 ml	0.5	1	0.5ml
3.	10 ml	1	1.4	0.4ml

(B)

S/No.	**Volume of water sample (ml) (V1)**	**Burette Initial readings**	**Burette Final readings**	**Volume of used AgNO3 soln.(ml)(V')**
1.	10 ml	1.4	11.2	9.8 ml
2.	10 ml	11.2	21	9.8 ml
3.	10 ml	21	30.7	9.7 ml

Calculation:

Volume of water sample for titration = 10ml

Volume of AgNO3(N/40) used = (V' -V) = 9.8 – 0.5

= 9.3ml

For Cl^{-} :

N1V1 = N2 V2

N1 x 10 = 1/40 x 9.3

N1 =1/400 x 9.3

N1=0.02325

Strength= N1 x Equivalent weight of Cl^{-}

= 0.02325x 35.5gm/l

= 0.825 gm/l

= 825ppm

Result:

Chloride content present in the given water sample is 0.825 gm/l or 825 ppm

Precautions:

1. The whole apparatus should be washed with distilled water before the start of the experiment.

2. The reaction mixture should be briskly shaken during the titration. 3. The end point of the reaction should be carefully observed.

4. The volume of the indicator should be same in all the titrations.

5. The pH of the sample solution should be adjusted to 7-8 ranges by adding acidic/basic solution.

6. Do not take mean of burette readings.

EXPERIMENT - 4

Object:

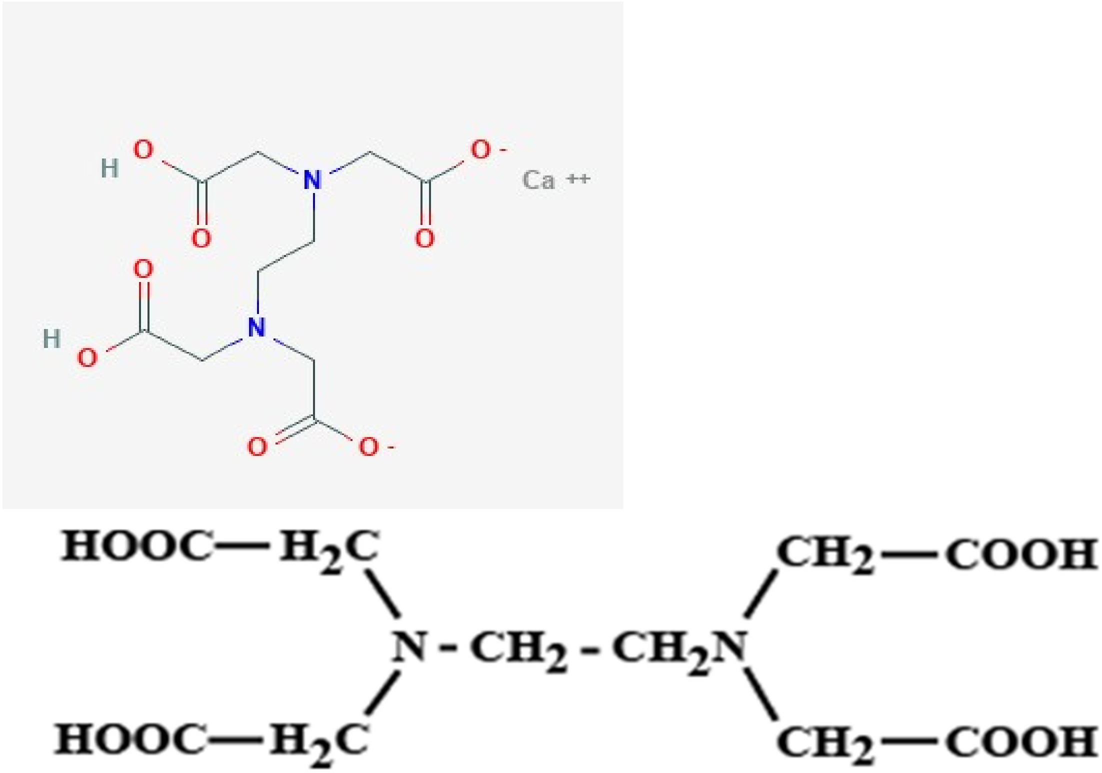
To determine the temporary, permanent and total hardness of water in a given sample of water by titrating it against standard EDTA solution (N/20) using Eriochrome Black-T as an internal indicator.

Materials/chemicals/apparatus required:

Burette, pipette, conical flask, beaker, measuring cylinder, tripod stand, wire gauze, funnel, filter paper, dropper, standard EDTA Solution (N/20), ammonium buffer solution, Eriochrome Black-T Indicator, Hard water (given water sample).

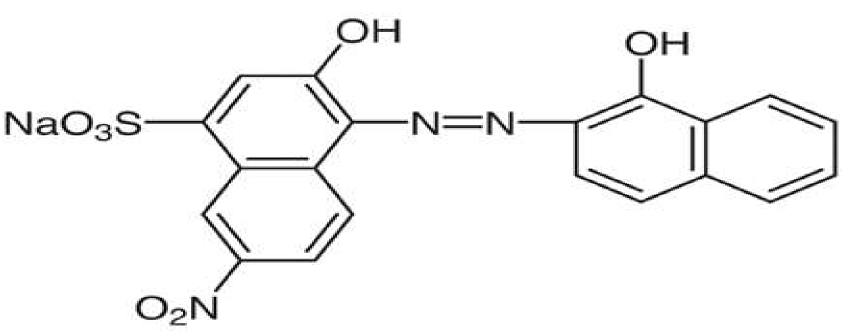
Principle:

When Eriochrome Black-T (indicator) is added to hard water solution at around 10.0 pH. it gives wine red colored unstable complex with $Ca^{2}+$ *and* $Mg2+$ *ions of the sample water. When this wine red colored complex is titrated against EDTA solution. The color of the complex changes from wine red to original blue color showing the end point. EDTA (Ethylene Diamine Tetra Acetic acid) is a well-known complexing agent, which is widely used in analytical work on account of its powerful complexing action and commercial availability.*



(Structure of EDTA)

EDTA complex with divalent metal cation (M = Ca^{2+} , $Mg2^{+}$) aqueous solution EDTA ionizes to give two Na^{+}ions and a strong chelating agent. The indicator used is a complex organic compound (sodium-1-(hydroxy-2-naphthylazo)-6 nitro-2 naphthol-4-salphonate) commercially known as Eriochrome Black-T. It has two ionisable phenolic hydrogen atoms and for simplicity it is represented as $Na^{+}H2$ Ion



(Structure of Eriochrome Black-T)Observation:

(A) For Hard Water:

S/No.	Volume of Hard water (ml)	Burette Initial readings	Burette Final readings	Volume of EDTA Solution used .(ml)
1.	10 ml	0.0	3.1	3.1ml
2.	10 ml	3.1	6.2	3.1ml
3.	10 ml	6.3	9.6	3.3ml

(B) For Boiled Water:

S/No.	Volume of water (ml)	Burette Initial readings	Burette Final readings	Volume of EDTA Solution used .(ml)
1.	10 ml	0	2.5	2.5ml
2.	10 ml	2.5	4.9	2.4ml
3.	10 ml	4.9	7.4	2.5ml

Calculation:

For Total hardness:

N1V1 = N2 V2

N1 x 10 = 1/20 x 3.1

N1 =1/200 x 3.1

N1=0.015N

Strength= N1 x Equivalent weight of Calcium Carbonate

= 0.015x 50gm/l

= 0.750gm/l

= 750ppm

For Permanent hardness:

N1V1 = N2 V2

$N1 \times 10 = 1/20 \times 2.4$

$N1 = 1/200 \times 2.4$

$N1 = 0.012N$

Strength= N1 x Equivalent weight of Calcium Carbonate

= 0.012x 50gm/l

= 0.6gm/l

= 600ppm

Temporary Hardness = Total Hardness – Permanent Hardness

= 750 – 600

= 150 ppm 0r mg/l

Result:

Total hardness= 750ppm

Permanent hardness=600ppm

Temporary hardness=150ppm

Precautions:

1. The glassware's should be properly rinsed with distilled water.

2. The reaction mixture should be shaken properly.

3. The end point should be noted correctly.

4. The pH should be maintained during titration.

5. The amount of indicator should be same in all titrations.

6. Do not take mean of burette readings.

EXPERIMENT – 5

Object:

To determine the content in the supplied sample of iron or by titrimetric analysis against standard potassium dichromate (N/20) solution using potassium ferricyanide $K3[Fe(CN)6]$ as an external indicator.

Materials/chemical/apparatus:

Standard $K2Cr2O7$ solution, potassium ferricyanide indicator, burette, Pipette, conical flask, glass rod, white glazed sile, iron sample (Mohr's salt)

Principle:

Potassium dichromate oxidizes ferrous sulphate present in Mohr's salt into ferric sulphate in presence of dilute sulphuric acid. In this titration potassium ferricyanide is used as an external indicator, which gives a greenish blue color due to the formation of ferro ferricyanide complex.

$$Cr_2O_7^{2-} + 14H^+ + 6e^- \rightarrow 2Cr^{3+} + 7H_2O$$

$$[Fe^{2+} \rightarrow Fe^{3+} + e^-] \times 6$$

$$Cr_2O_7^{2-} + 14H^+ + 6Fe^{2+} \rightarrow 2Cr^{3+} + 7H_2O + 6Fe^{3+}$$

$$Fe^{2+} + 2[Fe(CN)_6]^{3-} \rightarrow Fe_3[Fe(CN)_6]_2$$

(Ferro ferricyanide)

(Blue complex)

Observation:

S/No.	Volume of sample taken v1(ml)	Burette Initial Readings	Burette Final Readings	Color with the indicator	End point range	Volume of K2Cr2O7 Used v2(ml)

3. Do not take mean of burette readings.

1.	10	0 10	10 11	Blue Yellow	10-11	11
2.	10	0 10.8	10.8 10.9	Blue Yellow	10.8-10.9	10.9
3.	10	0 10.8	10.8 10.9	Blue Yellow	10.8-10.9	10.9

Calculation:

N1V1 = N2 V2

N1 x 10 = 1/20 x 10.9

N1 =1/200 x 10.9

N1=0.0545N

Strength= N1 x Equivalent weight of Fe^{2+}

= 0.0545x 56gm/l

= 3.052gm/l

= 3052ppm

Result:

The ferrous ion content in the supplied of iron ore is 3052 ppm or milligram/litre

Precautions:

1. Burette and pipette should be rinsed properly before starting the titration.

2. The volume of solution taken should maintain properly.

EXPERIMENT - 6

Object:

To determine the viscosity and surface tension of a given liquid (liquid of polystyrene) by using Ostwald viscometer and stalagmometer respectively. Viscosity of water is 0.0101poise and Surface tension of water is 72.14 dynes /cm at 25°C.

(A) For viscosity of the given liquid

Materials chemicals Apparatus required:

Ostwald viscometer, stop watch, R.D. Bottle, weight box, pipette, beaker, given liquid, water sample, etc.

Principle:

According to Poiseuill's Equation:

$\eta = \pi \rho r^4 t \;/\; 8vl$ *----------- eq.(1)*

$\rho = hdg$ *----------- eq.(2)*

Where, [η] =Viscosity, r-Radius, ρ-hydrostatic pressure, t= Time of flow of liquid, v-Volume of liquid, l-Path length

From eq. (1) & eq. (2) we get

$$\frac{\eta 1}{\eta 2} = \frac{d1}{d2} \times \frac{t1}{t2}$$

$d1$ *= density of water*

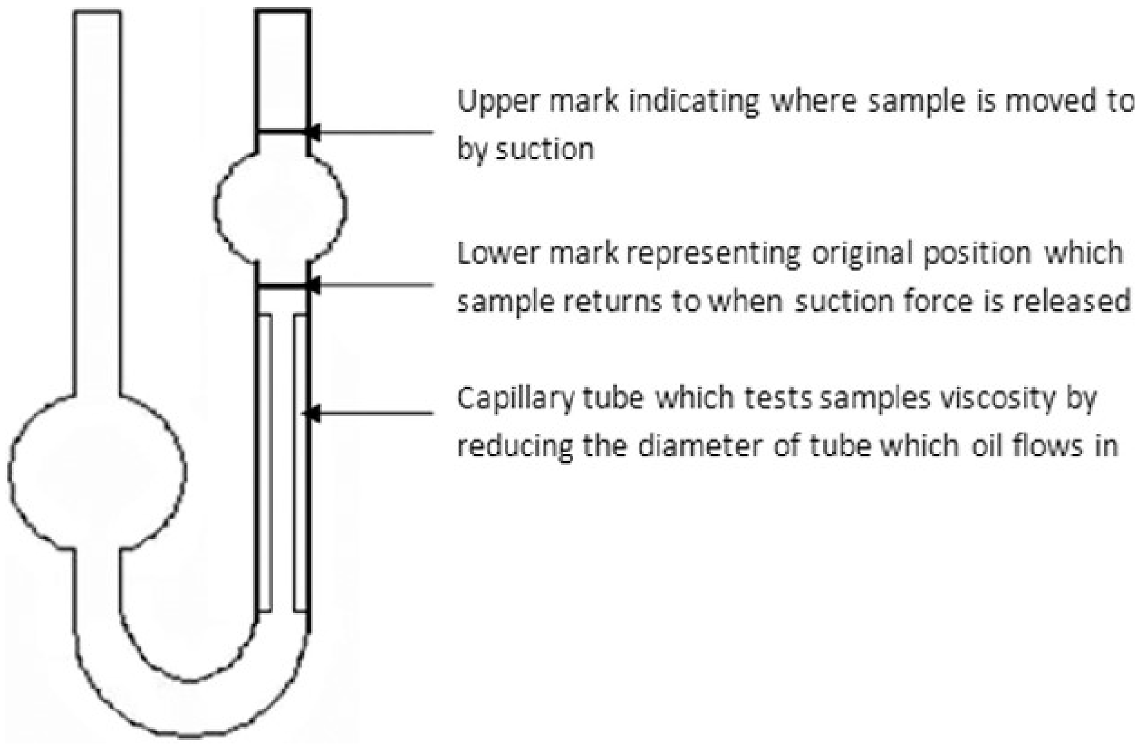
$d2$ *= density of liquid*

$t1$*= time flow of water*

$t2$*=time flow of liquid*

$\eta 1$*= viscosity of water*

$\eta 2$*= viscosity of liquid*



(Oswald Viscometer)

Observation:

(a)Table for time of flow:

S/No.	Time flow of liquid (sec)	Average Time flow of liquid(sec) (t2)	Time flow of water(sec)	Average Time flow of water(sec) (t1)
1.	93	93	64.2	64.2
2.	93.1		64.3	
3.	93		64.1	

(b)For density:

Weight of empty R.D. Bottle = 31.05gm

Weight of empty R.D. Bottle + water = 80.01gm

Weight of empty R.D. Bottle + liquid = 75.90gm

Weight of water = 48.96 gm

Weight of liquid = 44.85gm

Calculation:

$$\frac{\eta 1}{\eta 2} = \frac{d1}{d2} \times \frac{t1}{t2}$$

$$\frac{0.0101}{\eta 2} = \frac{48.96}{44.85} \times \frac{64.2}{93}$$

$$\eta 2 = 0.0134 \ poise$$

Result:

The viscosity of the given liquid sample = 0.0134 poise

Precautions:

(1) The number of drops and density of water/liquid should be measured carefully.

(2) The stalagmometer should be hanged vertically.

(3) No air bobble should be formed while sucking the water/liquid into the stalagmometer.