V.V.Vanniaperumal College for Women, Virudhunagar,Tamilnadu
No HRD-11011/163/2020-HRD-DBT/Chemistry /Analysis of Oil /Fats and Water

LAB MANUAL

ANALYSIS OF OIL / FATS AND WATER

(UNDER DBT STAR COLLEGE SCHEME)
Department of Biotechnology, Ministry of Science and Technology,
MHRD, New Delhi

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V.V.VANNIAPERUMAL COLLEGE FOR WOMEN
VIRUDHUNAGAR

V.V.Vanniaperumal College for Women, Virudhunagar,Tamilnadu
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V.V. VANNIAPERUMAL COLLEGE FOR WOMEN

(Belonging to Virudhunagar Hindu Nadars)
An Autonomous Institution Affiliated to Madurai Kamaraj University
Re-accredited with 'A' Grade (3rd cycle) by NAAC
VIRUDHUNAGAR – 626 001 (TAMIL NADU)

DBT STAR COLLEGE SCHEME

Ministry of Science and Technology,
Government of India, New Delhi

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FOREWORD

The Lab Manual for **"ANALYSIS OF OIL/FATS AND WATER"** is designed to acquaint the student with essential skills and techniques in accordance with the updated syllabus under DBT Star College Scheme sponsored by the Department of Biotechnology, Ministry of Science and Technology, MHRD, New Delhi. The skill of analysis and performing experiments will reinforce the theoretical knowledge of learnt concepts.

We thank the **Department of Biotechnology, The Ministry of Science and Technology, MHRD, New Delhi** for providing a good opportunity under Star College Scheme (No HRD11011/163/2020-HRD-DBT Dt. 24.08.2020). Under this scheme, we have purchased Chemicals and Glassware. This provision enables the students for better understanding of basic concepts in Chemistry and to develop curiosity for further progress.

We hope this manual surely fulfil the student's need to enhance their attitude towards research and empower them as a better chemist.

MEMBER SECRETARY/COORDINATOR **CHAIRMAN/PRINCIPAL**

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LIST OF PRACTICALS

1. DETERMINATION OF SPECIFIC GRAVITY OF AN OIL

CALCULATION:

Weight of empty specific gravity bottle	= W_1g
Weight of specific gravity bottle with water	= W_2g
Weight of specific gravity bottle with oil	= W_3g
Specific gravity of the given oil sample at °C	= $(W_2-W_1)/(W_3-W_1)$

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1. DETERMINATION OF SPECIFIC GRAVITY OF AN OIL

AIM: To determine the specific gravity of an oil using specific gravity bottle.

REQUIREMENTS:

EQUIPMENTS:	Digital balance
GLASSWARE :	specific gravity bottle of capacity 25 or 50ml, beaker

PRINCIPLE:

Specific gravity is an index used to measure the density of a liquid. It is the ratio of the density of the liquid to the density of water at 4°C.In the oil industry, specific gravity is a common quality specification for finished products.In specific-Gravity Bottle method, a flask is made to hold a known volume of liquid at a specified temperature (usually 20°C). From the weight measurements, specific gravity of various oils can be calculated.

DESCRIPTION:

The specific gravity bottle is a thin-walled pear-shaped, glass bottle with a narrow neck. A ground stopper tightly fits into the neck. A narrow orifice runs through the centre of the stopper. The bottle has usually a volume of 25 or 50 ml which is marked on its body. When the bottle is filled with a liquid completely without air bubbles and the stopper is applied, the liquid has this marked volume. The bottle must always be handled by the nook and not by the bulb, as the heat of the palm is likely to expand the liquid.

PROCEDURE:

Take the weight of empty specific gravity bottle accurately (W_1g). Fill completely with water and weigh it (W_2g). Dry it well,rinse it with the given oil and fill it completely with the given oil and take weight. Calculate the specific gravity of the oil as given below;

RESULT:

The specific gravity of the given oil sample is--------.

NO.OF BENEFICIARIES: 45

OUTCOME:

The students are able to determine the specific gravity of different oils.

2.DETERMINATION OF SURFACE TENSION

OBSERVATIONS:

1. Value of each pitch scale division=
2. Advance along pitch scale when head of screw is rotated 10 times =
3. Pitch of the screw =
4. No. of head scale divisions =
5. Least count =
6. Zero error (in head scale divisions)

Trial	Pitch scale reading	Head scale reading	Zero correction	Corrected H.S.R	Reading in mm
1.					
2.					
3.					

Mean:

The mean radius at the place may be taken as $\frac{r_1 + r_2}{2}$

2. DETERMINATION OF SURFACE TENSION

AIM: To determine the surface tension of an oil by drop weight method.

REQUIREMENTS:

EQUIPMENTS:	Digital balance,screw gauge
GLASSWARE :	Burette, a clear narrow tube, beaker, specific gravity bottle.

PRINCIPLE:

The principle is to measure the weight of drops of a fluid of interest falling from a capillary glass tube, and thereby calculating the surface tension of the fluid.

PROCEDURE:

A clean and dry narrow tube of about 4 to 5 mm in diameter is connected to the bottom of the burette by means of a rubber tube. The oil with the required surface tension is poured into the burette. A pinch clamp isapplied to the rubber tube and by working this the flow of the liquid from the burette into the tube can be so controlled that fully formed drops detach themselves from the tube. A beaker whose mass (m_1g) has already been determined is brought under the tube and a large number of drops, say 100, is collected in the beaker. The beaker is then weighed (m_2g). Then (m_2-m_1)g given the mass of 100 drops from which the mass (m g) of each drop is found. The external radius is found using a screw gauge (r).

Surface tension of oil (T) = mg/3.8 r(dynes /cm)

DETERMINATION OF EXTERNAL RADIUS USING SCREW GAUGE:

The given object is gently gripped between the tip of the screw and the stud, taking care not to press it too hard. The pitch scale reading is the total number of pitch scale divisions uncovered. The pitch of the screw is half mm. The pitch scale reading must also be judged correct to half mm.

The head scale reading is observed as the division on the head scale coinciding with the pitch scale. This reading is corrected and multiplied by the least count. The product is added to the pitch scale reading to get the correct thickness or diameter of the object. The experiment is repeated for various positions of object.

Pitch of the screw (mm) = $\frac{\text{number of divisions uncovered}}{\text{No. of revolutions}}$

Least count = $\frac{\text{pitch of the screw}}{\text{No. of division}}$

RESULT: The surface tension of the given oil sample is--------.

NO.OF BENEFICIARIES: 45

OUTCOME: The students are able to determine the surface tension of different oils.

3.DETERMINATION OF VISCOSITY

AIM: To determine the viscosity of the given oil sample

REQUIREMENTS:

EQUIPMENTS:	Digital balance,Ostwald's Viscometer, stop watch,thermostat
GLASSWARE :	pipette, capillary tube, cylindrical vessel.

PRINCIPLE:

There are two methods generally employed for the determination of the viscosity of a given oil;

(a) Ostwald's Viscometer
(b) Stroke's falling sphere method

A) OSTWALD'S VISCOMETER METHOD:

Ostwald's Viscometer is used for measuring viscosity in this method. It is thoroughly cleaned and dried.Usuallyknown volume(10 to 25ml) of water is pipetted out into the bulb C. It is then sucked into the bulb A through the capillary tube B with the help of a rubber tube attached at its end, till it rises to the mark x. Now the time taken by water to flow between the marks x and y is noted by means of a stop watch. Let it be t_1. The viscometer is dried and the process is repeated as before by taking the same of volume of the oil. Let the time of flow be t_2. Then evidently,

$$\eta_1 / \eta_2 = \rho_1 t_1 / \rho_2 t_2$$

whereρ_1 and ρ_2 are the densities of the two liquids whichcan be determined by specific gravity bottle method.Knowing the co- efficient of viscosity of water η_1 (1 centipoise at 20° C), that of oil η_2 can be calculated.

B) STROKES FALLING SPHEREMETHOD:

The liquid is taken in a tall cylindrical vessel and is maintained at a constant temperature using a thermostat (constant temperature bath). A small ball usually of stainless steel, is allowed to travel and the time taken is noted using a stop watch. The speed of fall can be calculated from the measured time and the distance between A and B.

Substitution of the measured values to the equation,

$$\eta = 2r^2(\rho_s - \rho_l)g / 9u$$

gives the value of η

r = radius of sphere (ball) (read shots)

a = uniform rate offall.

ρ_s = density of solid (ball)

ρ_1= density of oil.

g = acceleration due to gravity 980 cm/sec^2

In this method, the viscosities of two liquids can also be comparedby determining the speed of the ball in the two liquids one by one. The equation used for comparison is

$$\frac{n1}{n2} = \frac{(ps-p1)t1}{(ps-p2)t2}$$

Where ρ_1 and ρ_2 are the densities of two liquids respectively.

RESULT: The viscosity of the given oil sample is-------.

NO.OF BENEFICIARIES: 45

OUTCOME:

The students are able to determine the viscosity of different oils.

4. DETERMINATION OF PERCENTAGE OF FREE FATTY ACID IN AN OIL

TABULATION:

Titration between approx.N/10 sodium hydroxide solution and standard oxalic acid solution:

S.No.	Volume ofNaOH (ml)	Burette Reading(ml)		Volume of Oxalic acid(ml)	Concordant value	Indicator
		Initial	Final			
						phenolphthalein

CALCULATION:

% of FFA= $\frac{\text{Volume of NaOH x Normality x 0.282 x 100}}{\text{Weight of oil taken}}$

4. . DETERMINATION OF PERCENTAGE OF FREE FATTY ACID IN AN OIL

AIM: To determine the percentage of free fatty acid in an oil.

REQUIREMENTS:

EQUIPMENTS:	Digital balance
GLASSWARE :	Burette-50 ml, Pipette-20 ml, conical flask-250 ml, standard measuring flask-200 ml, funnel, weighing bottle
CHEMICALS:	Oxalic acid, Sodium hydroxide, phenolphthalein, rectified spirit

PRINCIPLE:

A small quantity of free fatty acids is usually present in oil along with the triglycerides.Thekeeping quality of oil therefore relies upon the free fatty acid content.The free fatty acid in oil is estimated by titrating it against NaOH in the presence of phenolphthalein indicator. All common oils like ground nut oil, gingelly oil, cotton-seedoil, rice bran oil except coconut oil contain oleic acid as free fatty acid. The equivalent weight of oleic acid is 28.

PROCEDURE:

PREPARATION OF STANDARD OXALIC ACID SOLUTION:

Weigh accurately 1.6 g of oxalic acid in a clean dry weighing bottle and transfer it into a 250ml SMF though a funnel. Dissolve it in minimum amount of water and then make with distilled water.

TITRATION BETWEEN AN APPROXIMATELY N/10 SODIUM HYDROXIDE SOLUTION AND STANDARD OXALIC ACID SOLUTION:

Fill the burette with oxalic acid solution. Pipette out 20ml of sodium hydroxide solution. Titrate it againstsodium hydroxide solution.The end point is the disappearance of pink colour. Repeat the titration to get concordant titre values. Calculate the strength of sodium hydroxide solution.

TITRATION BETWEEN THE OIL AND SODIUM HYDROXIDE SOLUTION:

Weigh accurately 10 g of oil in a clean dry conical flask (150ml) capacity. Add 50ml of rectified spirit and warm it. Add 2 drops of phenolphthalein. Titrate it against sodium hydroxide solution taken in the burette. The end point is persisting pink colour.The volume of sodium hydroxide isnoted and is used for the % of FFA calculation.

RESULT:

The percentage of free fatty acid in the given oil sample is---------.

OUTCOME: The students are able to determine the % of free fatty acid of the oil sample assessing the quality of the oil/fat sample.

5. DETERMINATION OF IODINE VALUE

TABULATION:

Titration between potassium dichromate solution and sodium thiosulphate solution

S.No.	Vol.of $K_2Cr_2O_7$(ml)	Burette Reading		Vol.of $Na_2S_2O_3$ (ml)	Concordant value	Indicator
		Initial	Final			
						Starch

CALCULATION:

Iodine number = $\frac{(V_1-V_2) \times N \times 126.9 \times 100}{W \times 1000}$

Whose V_1 is the volume of thiosulphate solution used for blank titration.

V_2 is the volumeof $Na_2S_2O_3$ solution used for the sample.

N is the normality of $Na_2S_2O_3$ solution.

W is the weight of the sample.

5. DETERMINATION OF IODINE VALUE

AI M: To determine the iodine value of the given oil sample

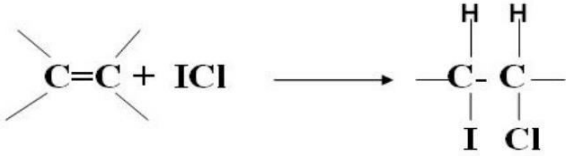
REQUIREMENTS:

EQUIPMENTS:	Digital balance
GLASSWARE :	Burette-50 ml, Pipette-20 ml, conical flask-250 ml, weighing bottle, standard measuring flask-50 ml,Erlenmeyer flask, Beaker, funnel
CHEMICALS:	Iodine monochloride, glacial acetic acid, potassium dichromate, sodium thiosulphate, starch, sulphuric acid, 10% KI solution

PRINCIPLE:

The most important application of the iodine value is to determine the amount of unsaturation contained in fatty acids. This unsaturation is in the form of double bonds which react with iodine compounds. The higher the iodine value, the more unsaturated fatty acid bonds are present in a fat.

Fatty acids react with a halogen [iodine] resulting in the addition of the halogen at the C=C double bond site. In this reaction, iodine monochloride reacts with the unsaturated bonds to produce a di-halogenated single bond, of which one carbon has bound an atom of iodine.



After the reaction is completed, the amount of iodine that has reacted is determined by adding a solution of potassium iodide to the reaction product.

$$2ICl + KI \text{ ----------> } 2KCl + I_2$$

This causes the remaining unreacted ICl to form molecular iodine. The liberated I_2 is then titrated with a standard solution of 0.1N sodium thiosulphate.

$$I_2 + 2\ Na_2S_2O_3 \text{ ----------> } 2\ NaI + Na_2S_4O_6$$

Saturated fatty acids will not give the halogenation reaction. If the iodine number is between 0-70, it will be a fat and if the value exceeds 70, it is an oil. Starch is used as the

indicator for this reaction so that the liberated iodine will react with starch to give purple coloured product and thus the endpoint can be observed.

PROCEDURE:

Iodine value or number is the measure of the amount of unsaturated fatty acids present in fats expressed as the number of grams of iodine absorbed by 100g of fat.

WIF'S METHOD:

In this method a solution of iodine monochloride in glacial acetic acid is used.(Wif's solution).

PREPARATION OF N/20 STANDARD POTASSIUM DICHROMATE SOLUTION:

Weigh accurately 0.5g of potassium dichromate in a clean dry weighing bottle and transfer it into a 200 ml standard flask through a funnel. Dissolve it in minimum water and then make up to the mark.

TITRATION BETWEEN POTASSIUM DICHROMATE SOLUTION AND SODIUM THIOSULPHATE SOLUTION:

Fill the burette with sodium thiosulphate solution. Pipette out 20 ml of dichromate solution into a clean conical flask. Add 10 ml of dil.H_2SO_4 and 15ml of 10% potassium iodide solution. Titrate the liberated iodine against sodium thiosulphate solution using starch as indicator. The end point is the change of colour from blue to green. Repeat the titration to concordant titre values. Calculate the strength of sodium thiosulphate solution.

TITRATION BETWEEN THE OIL AND WIF'S SOLUTION AD SODIUM THIOSULPHATE SOLUTION:

Weigh 0.3 g of oil accurately in a small beaker (10ml) and transfer it into an Erlenmeyer flask. Dissolve it in 10ml of carbon tetrachloride. Add 20ml (exactly) of Wif's solution. Stopper the flask. Shake it well and allow to stand for half an hour. At the end of the reaction period, add 15ml of 10% KI solution. Shake it well and titrate it against sodium thiosulphate solution using starch as indicator and note the volume of sodium thiosulphate solution as V_2. Blank titration is done adapting the same procedure without oil sample and noted as V_1.

RESULT:

The Iodine value ofthe given oil sample is---------.

IMPORTANT NOTE:

- Iodine monochloride is caustic. So handle the reagent with gloves.
- For better results, perform the experiments without any time gap during addition of reagents as the liberated iodine is susceptible to oxidation by light.

OUTCOME:

The students are able to determine the iodine number of the oil sample thereby can assess the quality of the oil sample.

6. DETERMINATION OF SAPONIFICATION VALUE

TABULATION:

Standardisation of N/2 HCl solution using standard Na_2CO_3 solution:

S.No.	Vol.of Na_2CO_3(ml)	Burette Reading(ml)		Vol.of HCl (ml)	Concordant value	Indicator
		Initial	Final			
						Methyl orange

CALCULATION:

Saponification value = difference in (V_1-V_2)m1x 56 xN/(Weight of the oil in g)

N= Normality of HCl.

6. DETERMINATION OF SAPONIFICATION VALUE

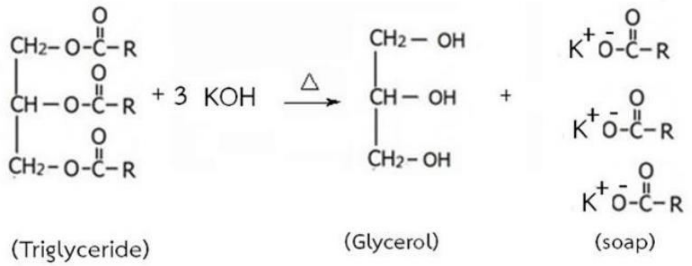
AIM: To determine the saponification valueof the given oil sample

REQUIREMENTS:

EQUIPMENTS:	Digital balance
GLASSWARE :	Burette-50 ml, Pipette-20 ml, conical flask-250 ml, standard measuring flask-100 ml, funnel, weighing bottle
CHEMICALS:	odium carbonate,alcoholic potash, neutral alcohol, hydrochloric acid, phenolphthalein

PRINCIPLE:

Saponification valueexpressed by number of milligrams of potassium hydroxide required to saponify 1 gm of the oil. The oil sample is saponified by refluxing with known excess of alcoholic potassium hydroxide solution.The alkali required for alkali saponification is determined by the titration of the excess potassium hydroxide with standard hydrochloric acid.



The saponification number is the index of the mean molecular weight of the fatty acids of glycerides comprising the fat. Lower the saponification number, larger the molecular weight of fatty acids in the glycerides and vice versa.

PROCEDURE:

PREPARATION OF N/2 SODIUM CARBONATE SOLUTION:

Weigh accurately 2.6 gm of sodium carbonate in a clean dry weighing bottle and transfer it into a 100ml standard flask through a funnel. Dissolve it minimum water and then make up to the mark.

STANDARDISATION OF N/2 HCl SOLUTION USING STANDARD Na_2CO_3SOLUTION:

Fill the burette with HCl. Pipette out 20ml of standard Na_2CO_3 into a clean conical flask and add 2 drops of methyl orange as an indicator into the conical flask. The solution turns yellow in colour. Titrate this against HCl solution in the burette. The end point is the change of colour from yellow to pale pink. Repeat the titration for concordant value. From the titre value, strength of sodium carbonate can be calculated.

TITRATION BETWEEN THE OIL AND ALCOHOLIC POTASH AND HCl:

Weigh accurately 2 g of the oil and transfer it into a saponification flask. Add 25ml of alcoholic potash solution and 25 ml of neutral alcohol (exactly). Connect the condenser and reflux it for 2-3 hours. Titrate it while hot against N/2 HCl using phenolphthalein as indicator. Note the volume of HCl as V_1.

BLANK TITRATION:

Pipette out 25ml alcoholic potash solution into a round bottomed flask. Rinse the pipette with 25ml of neutral alcohol. Connect the flask with a suitable condenser and reflux it for 2-3 hours.Titrate it while hot against N/2 HCl using phenolphthalein as indicator. Note the volume of HCl as V_2.

RESULT:

The Saponification value of the given oil sample is---------.

OUTCOME:

The students are able to determine the saponification number of the oil sample thereby can assess the quality of the oil sample.

7. ADULTERATION TESTS

AIM: To detect the presence of sesame oil as adulterant in thesamples of fats and oils.

REQUIREMENTS:

EQUIPMENTS:	Digital balance
CHEMICALS :	Hydrochloric acid,furfural, rectified spirit
REAGENTS :	2% solution of furfural
GLASSWARE :	: test tube

PRINCIPLE:

This test is used to detect the presence of added sesame oil, which is characterized by the presence of certain phenolic substances. The principle of the test is based on the reaction of phenols with furfurals in acid solution. The latter reacts with the phenols found in sesame oil to give a compound with a pink colour.

PROCEDURE:

Take 5ml of the oil in a test tube following add 5ml of HCl and 0.4 ml furfural solution. Shake vigorously for two minutes. Allow the mixture to stand for some time.

OBSERVATION AND INFERENCE:

The development of red or pink colour in the acid layer indicates the presence of sesame oil. Confirm by adding 5ml of water and shaking again. If the colour in acid layer persists, sesame oil is present. If the colour disappears it is absent.

NO OF BENEFICIARIES: 45

OUTCOME: The students are able to test the presence of sesame oil in ghee

8. HALPHEN'S TEST OR BAVAN'S TEST

AIM: To detect the presence of cottonseed oil as adulterant in thesamples of fats and oils.

REQUIREMENTS:

EQUIPMENTS:	Digital balance, water bath
CHEMICALS :	Sulphur, carbon di sulphide, cotton seed oil
GLASSWARE :	: test tube

PREPARATION OF REAGENT:

Sulphur Solution: Prepare 1% of sulphur in carbon disulphide and then add an equal volume of amyl alcohol

PRINCIPLE:

Cottonseed oil has unique types of fatty acids—malvalic and sterculic acids, which are called cyclopropenoid fatty acids. The test is also given by Hempseed oil, Kapok seed oil / oils and fats containing cyclopropenoid fatty acids. Hydrogenation and deodorization wholly or partially destroy the chromogens and react with diminished intensity. A positive reaction is not given by an oil heated to 250°C or above. The fat of animals fed on cottonseed meal (butter, lard) or other cottonseed products may give faint positive reaction by this test.

PROCEDURE:

Take about 5ml of oil in test tube and add it to an equal volume of the sulphur solution. Mix thoroughly by shaking and heat gently in the water bath (70°-80°C) for a few minutes with occasional shaking until the carbon disulphide has boiled off and the sample stops foaming. Place the tube in an oil-bath or a saturated lime-bath maintained at 110°-115°C and kept for 1 to 2 hours.

OBSERVATION AND INFERENCE:

The development of red colour on heating the oil with a solution of sulphur in carbon disulphide indicates the presence of cottonseed oil.

NO OF BENEFICIARIES: 45

OUTCOME: The students are able to test the presence of cottonseed oil in oils/fats.

9. ESTIMATION OF ALKALINITY IN WATER SAMPLE

TABULATION:

STANDARD SULPHURIC ACID VS WATER SAMPLE

Volume of water sample(ml)	Burette reading(ml)			Volume of sulphuric acid consumed (ml)	
	Initial	Final			
		P	M	P	M

CALCULATION

PHENOLPHTHALEIN ALKALINITY

Volume of sulphuric acid (phenolphthalein end point)= ..... ml

Strength ofsulphuricacid =.....N

Volumeofwater sample =…….ml (Concordant Value)

Phenolphthaleinalkalinity $= V_{acid} \times N_{acid} \times 50 \times 1000 / V_{watersample}$

= ------ppm

METHYL ORANGEOR TOTAL ALKALINITY

Volume of sulphuricacid (methyl orange end point) = ...ml

Strength of sulphuric acid = ... N

Volumeofwater sample =…….ml (ConcordantValue)

Methyl Orangealkalinity $= \frac{V_{acid} X N_{acid} X 50 X 1000}{V_{watersample}}$

= -----ppm

9. ESTIMATION OF ALKALINITY IN WATER SAMPLE

AIM:

Todeterminethep henolphthalein alkalinity and methylorangealkalinity inthegiven watersample.

REQUIREMENTS:

EQUIPMENTS:	Digital balance
CHEMICALS :	Sodium carbonate, sulphuric acid, phenolphthalein, methyl orange
GLASSWARE :	: Conical flask, pipettes, burette, funnel

PRINCIPLE:

Pure water is neutral in nature with pH7. Due to the presence of dissolved minerals in rainwater ,the pH increases and becomes alkaline.Alkalinity in water is due to the presence of hydroxide, carbonate and bicarbonate ions. The various alkalinities are estimated by titrating the water sample again stastandard acid using phenolphthalein and methyl orange indicators successively.

Hydroxide ions is completely neutralized to water using phenolphthalein indicator(single step neutralization). Carbonate ions is neutralized to bicarbonate ions using phenolphthalein indicator in the first step. In the second step, these bicarbonate ions is completely neutralized to water and carbon dioxide using methyl orange indicator (double step neutralization). Bicarbonate ions is completely neutralized to water and carbondioxide using methyl orange indicator(single step neutralization).

$$Na_2CO_3 + 2HCl \longrightarrow 2NaCl + CO_2 + H_2O$$

$$NaOH + HCl \longrightarrow NaCl + H_2O$$

Note

P = M	Presence of OH^- only
P = 0	Presence of HCO_3^-
P = ½M	Presence of CO_3^{2-}
P > ½ M	Both OH^- and CO_3^{2-} are present
P < ½ M	Both HCO_3^- and CO_3^{2-} are present

PROCEDURE:

PREPARATION OF STANDARD NA_2CO_3 SOLUTION:

About 0.53g of Na_2CO_3 is weighed accurately, transferred into a 100ml standard measuring flask, dissolved in distilled water and made up to the mark.

STANDARDISATION OF H_2SO_4 SOLUTION USING STANDARD NA_2CO_3 SOLUTION:

The burette is filled with H_2SO_4. Pipetted out 20ml of standard Na_2CO_3 into a clean conical flask. 2 drops of methyl orange is added as an indicator into the conical flask. The solution turns yellow in colour. It is titrated against H_2SO_4 solution in the burette. The end point is the change of colour from yellow to pale pink. The experiment is repeated for concordant value. The readings are noted in the tabular column

DETERMINATION OF ALKALINITY OF WATER SAMPLE:

Theburetteisfilledwithstandardsulphuricacidtothezerolevel.

20 ml of the given water sample is pipetted out into a conical flask. Twodropsofphenolphthaleinindicatorisaddedandtitratedagainstthestandardsulphuric acid. The end point is the disappearance of pink colour. The titre value isnoted. A drop of methyl orange indicator is added to the same solution after thephenolphthalein end point and the titration is continued until the solution becomesorange. The totaltitre value is noted. The titration is repeated to get concordantvalue,titre values arenoted and calculation is done.

RESULT AND INFERENCE:

The given water sample contains

Phenolphthaleinalkalinity= …….. ppm

Methylorangealkalinity = …….. ppm

SinceP……½M,……and…….ionsarepresentinthe givenwatersample.

NO. OF BENEFICIARIES: 45

OUTCOME:

The students are able to

- State and explain then principle of acid-base titration with special reference to the determination of alkalinity of a water sample.
- Prepare a standard solution of sodium carbonate.
- Standardise the given solution of sulphuric acid and use it in determining the alkalinity of water sample.
- compare the alkalinity of various water samples.

10. ESTIMATION OF SALINITY OF WATER SAMPLE

TABULATION:

Standardization of $AgNO_3$ solution using standard sodium chloride solution:

S.No.	Volume of NaCl (ml)	Burette Reading(ml)		Volume of $AgNO_3$(ml)	Concordant value(ml)	Indicator
		Initial	Final			
						Potassium chromate

Determination of salinity of water sample using standardized $AgNO_3$ solution:

S.No.	Volume of water sample(ml)	Burette Reading (ml)		Volume of $AgNO_3$ (ml)	Concordant value(ml)	Indicator
		Initial	Final			
						Potassium chromate

CALCULATION:

Chlorosity of water = $\frac{\text{Volume of } AgNO_3 \text{ consumed x Normality of } AgNO_3}{\text{Volume of the sample.}}$

Chlorinityof Water = Chlorosityof water/Density of water

The density of water is taken as 1g/ml

Salinity of water = 0.03 + (1.805 x chlorinity of water) = ------ppm

10. ESTIMATION OF SALINITY OF WATER SAMPLE

AIM: To determine the salinity of the given water sample

REQUIREMENTS:

EQUIPMENTS:	Digital balance
CHEMICALS :	0.05N $AgNO_3$, 5% potassium chromate
GLASSWARE :	: Conical flask, pipettes, burette, funnel

PRINCIPLE:

Salinity is generally defined as the salt concentration.The chloride ion concentration of water sample is determined by titration with silver nitrate. As the silver nitrate solution is slowly added, a precipitate of silver chloride forms. The end point of the titration occurs when all the chloride ions are precipitated.Potassium chromate can serve as an end point indicator for the argentometric determination of chloride ions by reacting with silver ions to form a brick-red silver chromate precipitate in the equivalence point region.

$Cl^- + AgNO_3 \rightarrow AgCl + NO_3^-$

$2AgNO_3 + K_2CrO_4 \rightarrow Ag_2CrO_4 + 2KNO_3$

PROCEDURE:

PREPARATION OF 0.05 N STANDARD SODIUM CHLORIDE SOLUTION:

0.05 N standard sodium chloride solution is prepared by accurately weighing 0.585g of sodium chloride, dissolving in distilled water and making up to the mark in a 200ml SMF.

STANDARDIZATION OF$AgNO_3$SOLUTION USING STANDARD SODIUM CHLORIDE SOLUTION:

The burette is filled with 0.05 N $AgNO_3$. 10 ml of standard sodium chloride solution is pipetted out into a clean conical flask and added few drops of potassium chromate solution and is titrated against $AgNO_3$. The end point is the appearance of brick red colour. The titration is repeated for concordancy.

DETERMINATION OF SALINITY OF WATER SAMPLE USING STANDARDIZED $AgNO_3$SOLUTION:

The burette is filled with 0.05 N $AgNO_3$. 10 ml of water sample is pipetted out into a clean conical flask and added few drops of potassium chromate solution. The water sample is titrated against $AgNO_3$. The end point is the appearance of brick red colour. The titration is repeated for concordancy.

RESULT: The salinity of the given water sample is---------- ppm.

NO. OF BENEFICIARIES:45

OUTCOME:

The students are able to

- Define the terms chlorinity and salinity of water samples
- Estimate the salinity of the water samples by volumetric method
- Relate the salinity of water to the life of the organisms
- Compare the salinity of various water samples.

11. ESTIMATION OF HARDNESS OF WATER

TABULATION:

Standardisation of EDTA solution using standard $CaCl_2$ solution:

S.No	Volume of $CaCl_2$(ml)	Burette Reading(ml)		Volume of EDTA(ml)	Concordant value(ml)	Indicator
		Initial	Final			
						Eriochrome black T

Determination of total hardness of the water sample:

S.No	Vol.of water sample(ml)	Burette Reading(ml)		Vol.of EDTA(ml)	Concordant value(ml)	Indicator
		Initial	Final			
						Eriochrome black T

CALCULATION:

Total hardness = vol.of (M/100) EDTA X 1000/Volume of waterppm (expressed as $CaCO_3$)

11. ESTIMATION OF HARDNESS OF WATER

AIM: To determine the total hardness of the given water sample

REQUIREMENTS:

EQUIPMENTS:	Digital balance
CHEMICALS :	Na_2-EDTA,NH_4Cl, NH_4OH, Eriochrome black-T indicator,$CaCO_3$,HCl(AR),triethanolamine, alcohol
GLASSWARE :	: Burette, pipette, conical flask, standard measuring flask

PRINCIPLE:

The hard water is defined as the water which does not lather well with soap.The lather production is inferred due to the presence of Cl^-,HCO_3^- , and $SO_4^{2-}$of Mg^{2+} and Ca^{2+} ions. So hardness determination involves the determination of $Ca^{2+}$and Mg^{2+} present in water sample. The total hardness is expressed as ppm/gallon of Ca^{2+} and Mg^{2+} salts together,calculated as $CaCO_3$. This is determined by titration with (M/100) Na-EDTA in buffered alkaline solution using Eriochrome black T solution as an indicator,since it forms chelates with Ca^{2+} and Mg^{2+}.

PROCEDURE:

PREPARATION OF M/10 $CaCl_2$SOLUTION:

1 g of $CaCO_3$ is weighed accurately and dissolved using little amount of Concentrated HCl. The contents are transferred into 100 ml SMF and made up to the mark.

STANDARDISATION OF EDTA SOLUTION USING STANDARD $CaCl_2$SOLUTION:

The burette is filled with approximately M/10 EDTA solution. 20 ml of $CaCl_2$ solution is pipetted into a clean conical flask. Diluted to 100 ml and heated to warm condition. 5 ml of NH_3-NH_4Cl buffer and a pinch of Eriochrome black T indicator is added. The colour is now pink, it is titrated with approximately (M/10) EDTA from a burette. The end point is the change of colour from wine red to blue. The titration is repeated for concordancy. From the volume of EDTA consumed, the strength of EDTA can be calculated.

Table shows rating of Total Hardness of water in ppm

Total Hardness of water in ppm	Rating
0-25	Very soft
25-50	soft
50-100	Moderately soft
100-150	Slightly hard
150-200	Moderately hard
200-300	Hard
300-600	Very hard
>600	Extremely hard

DETERMINATION OF TOTAL HARDNESS OF THE WATER SAMPLE:

100 ml of water sample is taken in a 250 ml conical flask and heated to warm condition.5 ml of NH_3-NH_4Cl buffer is added and a pinch of Eriochrome black T indicator is added. The colour is now pink, it is titrated with (M/100) EDTA from a burette. The end point is the change of colour from wine red to blue. The titration is repeated for concordancy.

RESULT: The total hardness of the given water sample is-------ppm.

NO.OF BENEFICIARIES: 45

OUTCOME:

The students are able to

- Understand the complex metric titration
- Rate the total hardness of the water samples