LABORATORY MANUAL FOR CHEMISTRY PRACTICALS

(21CYB101J)

(For first year M.Tech (Int.) students, common to all branches)

SRM INSTITUTE OF SCIENCE AND TECHNOLOGY



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I Instructions to the Students

- 1. Get yourself prepared by going through the procedure for the experiment for the day's work before coming to the laboratory.
- 2. Bring laboratory manuals, calculators and a piece of cloth for every practical class.
- 3. Wear a lab coat and shoes while working in the laboratory.
- 4. Use the minimum possible quantity of chemicals / reagents. Switch off the instruments, when they are not in use.
- 5. Handle the apparatus and glassware with utmost care. Keep the working table and sink neat and clean. Do not throw filter paper, broken bits of glasses on the table or into the sink.
- 6. After completion of the experiments, clean all the apparatus and keep them in their respective places. Replace the reagent bottles in the respective places at once after use.
- 7. Get the laboratory workbook corrected immediately after the completion of the experiment. Submit the record notebook before starting the next experiment.
- 8. Ensure that the details on experiments are recorded in the record of attendance and assessment at the end of each class.
- 9. Be punctual to the laboratory. Maintain discipline and cleanliness in the laboratory.
- 10. Report breakage immediately to the laboratory staff.
- 11. Switch off the instruments, when they are not in use.

II Introduction to Laboratory Experiments

Synthesis, analysis and characterization are important processes in chemistry. In synthesis compounds are prepared, its composition and structure can be determined through chemical analysis and characterization techniques. There are two general analytical methods: qualitative and quantitative analysis. Titrimetry is one of quantitative analysis. It involves the estimation in solution by measurement of volumes. It is a handy, quick and fairly accurate method of quantitative analysis. It is based on reactions between substances in aqueous solutions. It involves frequent measurement of volumes of the reacting solutions and is, therefore, called volumetric analysis. In volumetric analysis, two solutions are always made to react in a conical flask known as titration flask. One of these two solutions is taken in the burette and other in the conical flask. This process of finding out the volume of the titrant required to react completely with a known volume of the solution under analysis is known as **titration**.

The exact point at which a chemical reaction is completed during titration is called its **end point or equivalence point**. In order to find out the end point, a reagent is used either internally or externally which produces a visible change in colour just at the stage when the reaction is completed. Such a reagent is called an **indicator**.

Terms involved in Volumetric Analysis

Titration: The process of finding out the volume of one of the solutions required to react completely with the definite volume of the other solution is known as titration.

Standard solution (Titrant): In volumetric titration, one of the required solutions must be standard. A standard solution is the one whose strength (of concentration) is known. If a reagent is available in pure state, its standard solution is prepared by dissolving an accurately weighed amount of it in water and making the solution to known volume by dilution. It is called the **Direct Method.** All such substances whose standard solutions are made by the direct method are termed **Standard Substances** or **Primary standards.** Oxalic acid crystals,

succinic acid, anhydrous sodium carbonate, potassium dichromate, ferrous ammonium sulphate (Mohr's salt), silver nitrate, sodium chloride, potassium chloride etc. belong to this category. The following are the conditions for a chemical to be a primary standard.

The conduction for a chemical as a primary standard:

- 1. It must be obtained in a highly purified state.
- 2. It must be stable in the air.
- 3. It must be readily soluble in water.
- 4. It should possess a large equivalent weight so as to minimize the weighing error.
- 5. The error in determining the end point must be negligible.

The direct method is not possible in case of those substances which, in general, fall short of the above conditions e.g., alkali hydroxides, inorganic acids (like HCl, H₂SO₄, HNO₃, etc.,). These are called the **secondary standards**. Their standard solutions are prepared by the so-called indirect method. An approximately desired weight of the substance is dissolved in water and the solution is made up to a known volume. The exact strength of the solution is then determined by **Standardization**, i.e., titration with a suitable standard reagent.

End point and Equivalence point: The point at which the completion of reaction occurs is called equivalence point, whereas an end point is an observable physical change that occurs near the equivalence point.

Indicator: The substance which indicates the completion of the reaction by change in colour at the end point.

Strength: The amount of substances dissolved in one liter of a solution when expresses in grams is termed as strength of a solution. Strength of the solution can also be expressed in any of the following ways:

(i) Normality: It is the number of gram equivalents of the substance dissolved per liter of the solution. It is denoted by N,

N = Grams of solute per liter of the solution

Gram equivalent weight of the solute

If one gram equivalent of a substance is dissolved per liter of the solution, it is called Normal solution.

(ii) **Molarity:** It is the number of moles of the solute present per liter of the solution. It is denoted by M,

M = Grams of solute per liter of the solution

Molecular mass of the solute

(iii) **Molality:** It is the number of moles of the substance dissolved in 1000gm or 1kg of the solvent. It is denoted by m,

M = Grams of solute per 1000gm of the solvent

Molecular mass of the solute

Equivalent Weight

The equivalent weight of the substance is defined as the number of parts by the weight of it that combines with or displaces one part by weight of hydrogen or eight parts by weight of oxygen. Equivalent weight expressed in grams is called gram equivalent weight.

(i) Equivalent weight of an acid = Molecular weight of acid

Basicity of acid

(ii) Equivalent weight of a base = Molecular weight of base

Acidity of base

The expression which relates the normalities and volumes of the two-reacting solution in a titration is called Normality equation. It is expressed as,

$$N_1V_1 = N_2V_2$$

Where NI and V1 represent normality and volume used for one solution and N2 and V2 represent normality and volume used for the second solution.

Common glassware used in titrimetry is burette, pipette, measuring flask, measuring cylinder, conical flask and reagent bottles.

GENERAL PRECAUTIONS

All the apparatus (burette, pipette, conical flask, standard flask etc.,) should be washed first with water and then detergent followed by distilled water before use.

- 1. Rinse the burette with a solution to be taken in the burette.
- 2. Rinse the pipette with a solution to be measured in the pipette.
- 3. Do not rinse the conical flask with the solution taken in it. Wash with distilled water every titration.
- 4. After switching on the instrument allows some time for stabilization.
- 5. Calibrate the instrument whenever it is required.
- 6. At every instant, handle the instrument gently and register our observations with maximum sensitivity of the instrument.
- 7. Care should be taken to see that the electrodes of the cell are completely immersed in the solution before the reading is measured.
- 8. Immerse the cell/electrode always in a beaker containing distilled water, when they are not in use.

PRECAUTIONS TO BE TAKEN WHILE USING A PIPETTE

Pipette is a graduated tube with a bulb in the middle for measuring and transferring liquids from one vessel to another.

- 1. The pipette should be washed well before use and rinsed twice before filling it up with the solution to be measured.
- 2. Always use your fore-finger and not the thumb for closing its upper end.
- 3. The solution should not be sucked too fast as it may go inside the mouth.
- 4. Solutions of poisonous substances should not be sucked with mouth-instead use a pipette filler or burette.
- 5. Do not blow the last drop remaining in the pipette.
- 6. Always keep the pipette in a vertical position/vertically.
- 7. Hot solutions should not be measured using a pipette.

PRECAUTIONS TO BE TAKEN WHILE USING A BURETTE

- 1. Burette is a graduated glass tube, commonly having a stop-cock at the bottom and is used for measuring liquids.
- 2. The burette should be washed before usage should be rinsed twice before filling with the solution to be measured
- 3. While taking a reading, the graduation corresponding to the lower meniscus of the solution should always be read for colorless solution.
- **4.** The reading should be taken by keeping the eye at the same level as that of the meniscus and as close to the level as possible to avoid error due to parallax.

Standard (Volumetric) flasks

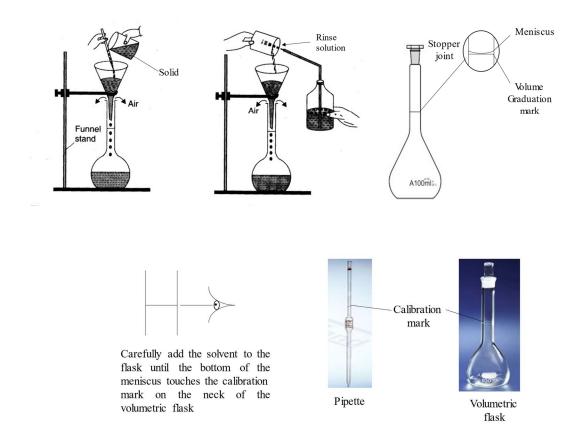
Standard flasks have a flat bottom, pear shaped body and a long narrow neck. These are usually calibrated to contain the specified volume when filled to the lined etched on the neck, and are used in the preparation of standard solutions and in the dilution of given solution to known volumes. The flasks most commonly used have capacities of 25,50,100,250,500 and 1000mL.

Making a given concentrated solution to a known volume in a standard measuring flask

To make up the given solution to the specified volume, the solution is carefully transferred with the help of a thin glass rod and a small funnel. Care should be taken so that not even a fraction of a drop of the given concentrated solution is wasted by split or any other means. For this purpose, the given solution is handled in the following way.

First wash the mouth of the solution-bottle with a little quantity of distilled water. Then transfer the whole solution in a thin stream to the flask through a small funnel using a glass rod. While pouring the solution, it should be observed that the solution is readily collected in the flask and does not form a film on the surface of the funnel. The solution-bottle has to be washed at least four times with a little quantity of distilled water at a time. The collected solution is mixed further with water and thoroughly shaken to get a uniformLy concentrated solution. Rest of the volume in the flask is slowly filled up with water coming as a line (stream) from a wash bottle. When the volume of the solution is nearing the glass mark, a clean pipette can also be made use of. Water should be added in drops, at the same time observing that the liquid meniscus level coincides with the mark. For the dilution of colorless solutions, the lower meniscus coincidence is taken as the making up mark.

On completion of the make-up process, the graduated flask is stoppered and shaken several times by placing the stopper in the middle of the left-hand palm and holding the bulb of the flask in the right hand. Invert the flask and shake. This procedure ensures a well uniformLy concentrated solution. Never remake the solution to the mark after shaking even if the liquid level is seen below the mark. This may be due to a thin film formation or sticking up of a fraction of drops in the neck of the flask.



Data Analysis

In every measurement all efforts should be made to minimize the uncertainty, eliminate errors and maximize reliability of data. Personal judgement and the soundness of the principle of the experiment chiefly determine the accuracy of data. In science, incorrectly recorded results are worse than no results at all. Incorrect results lead to wrong calculations. Scientific data are subjected to certain mathematical and statistical tests before accepting and reporting.

Precision

Precision is defined as the agreement between the numerical values of two or more measurements of the same object that have been made in an identical manner. Thus, a value is said to be precise when there is agreement between a set of results for the same quality. However, a precise value need not be accurate.

Accuracy

Accuracy represents the nearness of a measurement to its expected value. Any difference between the measured value and the expected value is expressed as the error. The difference between accuracy and precision must be noted. Accuracy is nearness to a true or expected value whereas precision is concerned with agreement among the measurements made in the same way.

III Rubrics for Evaluation of the Experiments

0.37	37 0.1	1.6.1.701	36 1 1 1	 1
S.No	Name of the	Marks (Observation/	Marks based	Total
	experiment	Calculation / graph)	on Percentage	Marks to be
			error	awarded for
				an
				experiment
1.	Determination of the			
	amount of sodium			
	carbonate and			
	sodium hydroxide in			
	a mixture by			
	titration.			
2.	Determination of			
	strength of an acid			
	by Conductometry.	6	0-1% = 4	10 marks
3.	Determination of		Marks	
	hardness (Ca ²⁺) of		1.1-2% = 3	
	water using EDTA –		Marks	
	Complexometric		2.1-3% = 2	
	method.		Marks	
4.	Determination of		3.1-4% = 1	
	ferrous ions using		Mark	
	potassium		Above 4.1 %	
	dichromate by		= 0	
	Potentiometric			
	titration.			
5.	Estimation of the			
	amount of chloride			
	content of a water			
	sample.			
6.	Determination of			
	molecular weight of			
	polymer by viscosity			
	average method.			
7.	Determination of the			
	strength of a mixture			
	of acetic acid and			
	hydrochloric acid by			
	Conductometry.			
8.	Determination of			
	strength of an acid			
	using pH meter.			

IV LIST OF EXPERIMENTS

- 1. Determination of the amount of sodium carbonate and sodium hydroxide in a mixture by titration.
- 2. Determination of strength of an acid by conductometry.
- 3. Determination of hardness (Ca²⁺) of water using EDTA complexometric method.
- 4. Determination of ferrous ions using Potassium dichromate by potentiometric titration.
- 5. Estimation of the amount of chloride content of a water sample.
- 6. Determination of molecular weight of polymer by viscosity average method.
- 7. Determination of the strength of a mixture of acetic acid and hydrochloric acid by conductometry.
- 8. Determination of strength of an acid using pH meter.

1.Determination of the amount of Sodium Carbonate and Sodium Hydroxide in a Mixture by Titration

EXPERIMENT NO. DATE:

AIM:

To determine the amount of sodium carbonate (Na₂CO₃) and sodium hydroxide (NaOH) in a mixture using hydrochloric acid.

CHEMICALS REQUIRED:

Sodium carbonate (Na₂CO₃), Sodium hydroxide (NaOH), Hydrochloric acid (HCl), Phenolphthalein, Methyl orange, Distilled water.

PRINCIPLE:

The titration of a mixture of NaOH and Na₂CO₃ with a standardized HCl solution has two equivalence points. The first equivalence point is due to the conversion of all the carbonate to bicarbonate and sodium hydroxide to sodium chloride as follows:

The pH of the resulting solution is around therefore phenolphthalein could be used as an indicator. The second equivalence point is due to the reaction of the resulting bicarbonate with an excess of HCl solution as follows:

$$NaHCO_{3} + HCl \rightarrow NaCl + CO_{2} + H_{2}O$$

In this stage methyl orange is used to find the equivalence point because the pH of the solution at this point will be around 3.8.

PROCEDURE:

Titration I: Standardization of HCL

Pipette out 20mL of standard sodium bicarbonate solution in a 250mL clean conical flask. Add 2-3 drops of methyl orange indicator. The solution will turn yellow in colour. Titrate the mixture against hydrochloric acid taken in the burette. The end point is the colour change from yellow to orange. Repeat the titration for the concordant values.

Titration II: Estimation of the mixture of (Na₂CO₃+NaOH)

Make up the given solution in a 100mL standard measuring flask. Pipette out 20mL of the made-up solution in a 250mL conical flask. Add 2-3 drops of phenolphthalein indicator. The solution is titrated against standard hydrochloric acid in a burette. The end point is the colour change from pink to colorless. Titre value is noted as AmL. to the same conical flask solution 2-3 drops of methyl orange indicator was

added. (Without pouring out the conical flask solution) The solution will turn yellow. This is titrated against standard hydrochloric acid from

"A" mL till "B" mL is reached. That is till end point orange colour is reached. The titration is repeated for the concordant values.

Titration I: Standardization of HCl

S.No.	Volume	of	pipette	Burette	reading	Concordant value	Indicator
	solution (1	mL)		(mL)		(mL)	
	(Na ₂ CO ₃	soluti	on)				
				Initial	Final		
							Methyl
							Orange

Calculation:

Volume of HCl $(V_2) =$

Normality of HCl $(N_2) =$

Volume of Na_2CO_3 $(V_1) = 20 \text{ mL}$

Normality of Na_2CO_3 (N_1) = 0.05 N

$$V_1N_1=V_2N_2$$

$$N_2 = (V_1 * N_1) / V_2$$

=....N

Titration II: Estimation of the mixture of Na₂CO₃ and NaOH

S.No.	Volume of pipette	Burette	Burette reading		Concordant value		Indicators used
	solution (mL)	(mL)	(mL)		(mL)		
	(Na ₂ CO ₃ +NaOH)	Initial	F	inal			
			HPh	M.O	HPh	M.O	
					(A)	(B)	
							Phenolphthalein(Hph)
							Methyl Orange(M.O)

A – Phenolphthalein End point – Volume of the acid used up to HPh end point

B – Methyl Orange End point – Total volume of acid used till M.O end point

(B-A) = C – Volume of acid used for $\frac{1}{2}$ CO₃²-neutralisation

2C-Total volume of acid used for complete neutralisation of CO_3^{2-} ions

Calculations:

Estimation of Na₂CO₃

Volume of HCl $(V_1) = 2C$

Normality of HCl (N_1) = from titration I

Volume of mixture $(V_2) = 20mL$

Normality of mixture $(N_2) = ?$

$$V_1N_1=V_2N_2$$

$$N_2 = (V_1 * N_1) / V_2$$

=.....N

Amount of Na₂CO₃ present in the whole of

= (Normality

× (Equivalent weight of

the given solution

of Na₂CO₃)

 Na_2CO_3

$$= (N_2*53)/10$$

=.....gm/L

Estimation of NaOH:

Volume of HCl

$$(V_1) = A - C (mL)$$

Normality of HCl (N_1) = from titration I

Volume of mixture $(V_2) = 20mL$

$$(V_2) = 20 \text{mL}$$

Normality of mixture $(N_2) = ?$

$$V_1N_1 = V_2N$$

 $N_2 = (V_1*N_1)/V_2$

Amount of NaOH present in the whole of the given solution

× (Equivalent weight of

of NaOH) NaOH)

10

$$=$$
 $N_2 \times 40$

10

RESULT:

Amount of NaOH present in the 100mL of the given solution = ____ gm/L Amount of Na_2CO_3 present in the 100mL of the given solution = _____ gm/L Bottle No. = ____

Rubrics for Evaluation	Marks
Experiment, Observation, Calculation	
% of error	
Signature of the teacher with date	

2. Determination of strength of an acid by conductometry

EXPERIMENT NO. DATE:

AIM:

To determine the strength of a given HCl solution with standard NaOH by conductometric titrations.

APPARATUS REQUIRED:

Burette, Pipette, Standard measuring flask, glass rod, Beaker, Wash bottle, funnel, Conductivity meter, conductivity cell.

CHEMICALS REQUIRED:

NaOH solution (0.1N), distilled water, HCl solution whose concentration is to be found.

PRINCIPLE:

The principle of the conductometric titration is that during a titration process, one ion is replaced with another and the difference in the ionic conductivities of these ions directly impacts the overall electrolytic conductivity of the solution. It is evident from the equation that as NaOH solution is gradually added, the H⁺ ion having high ionic conductance are replaced by Na⁺ having lower ionic conductance and hence the conductivity of the solution gradually decrease. At the equivalent point the conductivity would be minimum due to replacement of all H⁺ ion by Na⁺ ion. After the equivalence point Na⁺ and OH⁻ would accumulate in the solution and conductance of the solution will again increase

$$H^{+} + Cl^{-} + Na^{+} + OH^{-} \rightarrow Na^{+} + Cl^{-} + H_{2}O$$

PROCEDURE:

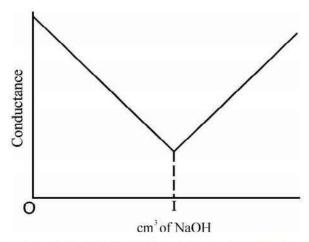
Make up the given HCl solution in a 100mL standard measuring flask. Pipette out 10mL of the made up HCl into a beaker. Dilute the solution with distilled water, so that the conductivity cell can be immersed well in the solution (up to 100mL). Stir the solution well with the help of a glass rod. Note down the conductance of the solution from the conductivity meter. Fill the burette with standard NaOH solution and run down into the beaker in small increments (1mL) with gentle stirring of the contents of the beaker.

After each addition, stir the contents of the beaker and after an equilibrium time of 2-3minutes note the corresponding conductance value and tabulate it. Continue the titration up to 15 increments, after the conductance reaches a minimum and starts increasing after the completion of the titration, wash the conductivity cell with distilled water and immerse in distilled water.

Plot a graph between conductivity and volume of NaOH added. The intersection of two lines to the volume axis gives the end point.

Volume of NaOH required for neutralization is taken from graph (fair) on intersection point which is corresponding to the volume axis (x). In order to get accurate results, perform a fair titration by adding NaOH in small amounts (0.2mL) near and beyond the end point. Calculate the strength of the given strong acid from the given NaOH strength.

MODEL GRAPH:



Conductometric titration of a strong acid (HCl) vs. a strong base (NaOH)

TITRATION 1:

Titration between standard NaOH and unknown HCl (pilot)

S.No	Volume of NaOH (mL)	Conductance (mho)

TITRATION 2:

Titration between standard NaOH and unknown HCl (fair)

S.No	Volume of NaOH (mL)	Conductance (mho)

CALCULATION:

 $\label{eq:V1} \begin{array}{ll} \mbox{Volume of HCl solution} & = V_1 & \mbox{$^{(10mL)}$} \\ \mbox{Normality of HCl solution} & = N_1 \\ \mbox{Volume of NaOH} & = V2 \mbox{ (from the 2nd graph)} \\ \mbox{Normality of NaOH} & = N_2 \mbox{ (0.1N)} \\ \mbox{$V_1N_1 = V_2N_2$} \\ \mbox{$N_1 = V_2N_2/V_1$} \end{array}$

The strength of the given HCl solution = -----N

The strength of the given HCl solution =	N
Bottle number =	

Rubrics for Evaluation	Marks
Experiment, Observation, Calculation and	
Graph	
% of error	
Signature of the teacher with date	

3. Determination of Hardness (Ca²⁺) of Water Using EDTA – Complexometric Method EXPERIMENT NO. DATE:

Aim

To estimate the amount of total hardness, permanent hardness and temporary hardness of a given water sample by EDTA method.

Apparatus Required

Burette, Pipette, Conical flask, Standard volumetric flask, funnel.

Reagents Required

Standard hard water, sample water, boiled water, Eriochrome Black-T (EBT) indicator, ammoniacal buffer (NH₃-NH₄Cl) solution and ethylenediamine tetra acetic acid (EDTA).

Principle

Ethylenediamine tetra acetic acid disodium salt is used to determine the total hardness, permanent hardness (sulphate and chlorides of calcium & magnesium) and temporary hardness (bicarbonates of calcium & magnesium) of the given water sample. The hardness causing metal ions (Ca²⁺ & Mg²⁺) form wine-red coloured weak complex with Eriochrome Black-T indicator in the presence of a buffer solution. The indicator is replaced by EDTA and a stable complex is formed with the addition of EDTA. Due to the removal of EBT indicator, wine-red colour changes to steel blue. The entire reaction between metal ions and EBT is represented as follows

- 1. Ca²⁺/ Mg²⁺+EBT ----- [Ca/Mg EBT] [Unstable complex, wine red] The unstable complex is quickly replaced by Ca/Mg EDTA complex at pH 9-10
- 2. Ca/Mg EBT +EDTA ----- \rightarrow Ca/Mg EDTA [Stable complex] + EBT [Blue]

Procedure

Titration I: Standardization of EDTA

Pipette out 20 mL of standard hard water into a clean conical flask. Add 5 mL of ammoniacal buffer solution followed by 2 to 3 drops of EBT indicator in to the conical flask. The solution turns into wine red colour immediately. Titrate the wine-red coloured solution against EDTA taken in the burette. The change of wine-red colour to steel blue colour is observed as the end point. Repeat the titration for concordant values. Let the titre value be noted as V₁ mL.

Titration II: Determination of Total Hardness

Pipette out 20 mL of sample water into a clean conical flask. Add 5 mL of ammoniacal buffer solution and 2 to 3 drops of EBT indicator. Now the solution turns into wine red in colour. Titrate the wine-red coloured solution against EDTA taken in the burette. The change of wine-red colour to steel blue colour is end point. Repeat the titration for concordant values. Let the titre value be noted as V_2 mL.

Titration III: Determination of Permanent Hardness

Take 100 mL of the water sample in a 500 mL beaker and boil gently for about one hour. Cool and then filter it into a 100 mL standard flask and make the volume up to the mark. Pipette out 20 mL of boiled water into a clean conical flask. Add 5 mL of ammoniacal buffer solution and 2 to 3 drops of EBT indicator. The solution turns wine red in colour. Titrate the wine-red coloured solution against EDTA taken in the burette. The change of wine-red colour to steel blue colour is the end point of the titration. Repeat the titration for concordant values. Let the titre value be noted as V₃ mL.

Titration I: Standardization of EDTA

Table 1: Standard hard water Vs EDTA

S.No.	Vol. of std. hard	Burette Reading		Vol. of EDTA	Indicator
	water (mL)	Initial (mL) Final (mL)		(mL)	
					EBT

Titration II: Determination of Total Hardness

Table 2: Sample water Vs EDTA

S.No.	Vol. of Sample	Burette l	Reading	Vol. of EDTA	Indicator
	water (mL)	Initial (mL)	Final (mL)	(mL)	
					EBT

Titration III: Determination of Permanent Hardness

Table 3: Boiled water Vs EDTA

S.No.	Vol. of boiled	Burette l	Reading	Vol. of EDTA	Indicator
	water (mL)	Initial (mL) Final (mL)		(mL)	
					EBT

Calculation

Titration – I

1 mL of standard hard water contains 1 mg of CaCO₃

Therefore, 20 mL of standard hard water contains 20 mg of CaCO₃

20 mL of standard hard water consumes V₁ mL of EDTA (or)

V₁ mL of EDTA is equivalent to 20 mg of CaCO₃

Hence, 1 mL of EDTA is equivalent to (20/V₁) mg of CaCO₃

Titration - II

V2 mL of EDTA is equivalent to (V_2/V_1) X 20 mg of CaCO₃ equivalent 20 mL of sample water will contain = (V_2/V_1) X 20 mg of CaCO₃ equivalent; Therefore, 1000 mL of the sample water will contain = (1000/20) X (V_2/V_1) X 20 mg of CaCO₃ The total hardness of the given water sample = _____ppm.

Titration - III

V3 mL of EDTA is equivalent to (V_3/V_1) X 20 mg of CaCO₃ equivalent 20 mL of boiled water will contain (V_3/V_1) X 20 mg of CaCO₃ equivalent; Therefore, 1000 mL of the boiled water will contain = (1000/20) X (V_3/V_1) X 20 mg of CaCO₃ The permanent hardness of the given water sample = ____ppm.

The temporary hardness of the given water sample

Total hardness—Permanent hardness

=.	• • • • • • • • • • • • • • • • • • • •	ppm
Result		
The total hardness of the given water sample	=	ppm
The permanent hardness of the given water samp	ple =	ppm
The temporary hardness of the given water samp	ple =	ppm

Rubrics for Evaluation	Marks
Experiment, Observation, Calculation	
% of error	
Signature of the teacher with date	

4. Determination of ferrous ions using Potassium dichromate by potentiometric titration

DATE: EXPERIMENT NO.

Aim:

To estimate the amount of ferrous (Fe²⁺) ions present in the given solution.

Apparatus Required:

Potentiometer assembly, 25mL burette, 10mL pipette, 250mL beakers, standard flask, calomel and

platinum electrodes.

Reagents Required:

Ferrous ammonium sulphate, dil. H₂SO₄, Standard K₂Cr₂O₇

Principle:

Measurement of EMF of an electrochemical cell using a potentiometer and change in EMF due to the

chemical (redox) reaction is monitored. In this potentiometric titration set up, an indicator electrode

(Pt electrode) and reference (calomel) is coupled to form electrochemical cell for Fe²⁺ to Fe³⁺. Fe²⁺ is

oxidizes to Fe³⁺ as K₂Cr₂O₇ is progressively added. Platinum electrode which is kept in contact with a

mixture of Fe²⁺ and Fe³⁺ ions act as a redox electrode (indicating the redox reaction). The reduction

potential of this single electrode depends on the ratio of [Fe²⁺/Fe³⁺] initially. During the titration of

Fe²⁺ in H₂SO₄ medium with K₂Cr₂O₇, this ratio varies to a little extent at the beginning and suddenly

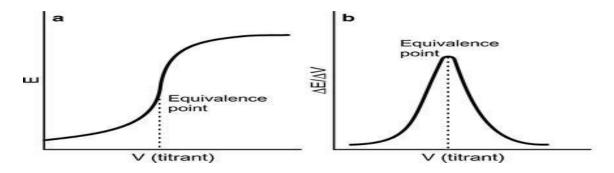
near the end point. It is observed that there is a sudden increase in the EMF of the cell at equivalence

point.

The cell set up: Hg| HgCl₂(s), KCl (1N) \parallel Fe²⁺|Fe³⁺, Pt

The chemical reaction: $6Fe^{2+} + Cr_2O_7^{2-} + 14H^+ \rightarrow 6Fe^{3+} + 2Cr^{3+} + 7H_2O_7^{2-}$

MODEL GRAPH:



Procedure:

The given ferrous iron solution is made up to a known volume (say 100mL) in a SMF following the standard procedure with usual precautions. Exactly 10mL of the made up Fe²⁺ solution is pipette out into a clean 100mL beaker. About 10mL of dil.H₂SO₄ and 100mL of distilled water are added to it. A platinum electrode is dipped into this solution and coupled with the standard calomel electrode. The resultant cell is then incorporated into the potentiometric circuit. Standard K₂Cr₂O₇ solution, which is taken in a burette, is added in installments of 1mL into the beaker and the cell EMF is measured after addition by proper mixing. Continue the process till and also well beyond the neutralization point which is indicated by abrupt change in the EMF. Note the volume of K₂Cr₂O₇ solution required for complete oxidation of Fe³⁺ solution from the plot of EMF versus the volume of the standard K₂Cr₂O₇ solution added. Plot the graph by taking Emf Vs volume of K₂Cr₂O₇ addition. The end point is taken from the mid-point of S- shape graph.

Perform similar titration by adding 0.1mL portions of added $K_2Cr_2O_7$ solution till there is an abrupt change in the EMF is observed. Tabulate the measured EMF corresponding to each addition of $K_2Cr_2O_7$. Plot the graph by taking $\Delta E/\Delta V$ vs volume of $K_2Cr_2O_7$ addition. The exact end point can be determined from the peak value meeting the x-axis from the second graph.

Table 1: Pilot titration: (FAS Vs K₂Cr₂O₇)

S. No.	Volume of K ₂ Cr ₂ O ₇ (mL)	EMF (Volts)	ΔE (Volts)
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			

Table 2: Fair Titration (FAS Vs K₂Cr₂O₇)

S.	Volume of	EMF	ΔE (volts)	ΔV (mL)	ΔΕ/ ΔV
No.	K ₂ Cr ₂ O ₇	(volts)			(Volts/mL
	(mL))
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					

CALCULATION:

Volume of pipette solution (FAS) = V_1 mL

Volume of $K_2Cr_2O_7 = V_2 mL$

Normality of $K_2Cr_2O_7 = N_2$

Normality of FAS $(N_1) = ?$

Strength of FAS = $N_1 = \underline{V}_2 \underline{N}_2$

 V_1

Amount of $Fe^{3+}(gm/Lit) = Eq.$ wt.* normality of Fe^{3+}

Amount of Fe ²⁺	in 100mL	= Normality	of Fe ²⁺ *	55.85
		•		

RESULT:
KESULI.

The amount of iron present in the given solution =gm/L
Bottle number =

Rubrics for Evaluation	Marks
Experiment, Observation, Calculation and	
Graphs	
% of error	
Signature of the teacher with date	

5. Estimation of the amount of chloride content of a water Sample

EXPERIMENT NO. DATE:

Aim:

To estimate the amount of chloride content in a water sample by Mohr's method.

Apparatus required:

Burette, Pipette, Conical flask, Glass rod, Funnel, Standard flask.

Reagents required:

1.05 N NaCl solution, AgNO₃ Solution, 2% K₂CrO₄ indicator, DI water, Test solution

Principle:

It is an example of precipitation reaction. The reaction between chloride and silver nitrate is direct and simple. It proceeds as follows:

$$AgNO_3 + NaCl \rightarrow AgCl \downarrow + NaNO_3$$

The completion of the reaction in this case is observed by employing K₂CrO₄ solution as the indicator. At the end point, the yellow colour of solution change into reddish brown due to the reaction

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

K₂CrO₄ indicator will not be precipitated as Ag₂CrO₄ until all the chlorides in the solution have been precipitated as AgCl.

Procedure:

Titration I- Standardization of Silver Nitrate solution

Pipette out 20mL of standard NaCl solution (0.05 N) into a clean conical flask. Add 1mL of 2% K₂CrO₄ indicator solution (The solution turns yellow in color). Titrate it against the AgNO₃ solution taken in the burette. Shake the conical flask continuously and continue to titrate until the reddish-brown tinge of the solution does not disappear. Note down the burette reading and repeat the experiment for concordant values.

Titration II: Estimation of Chloride

Transfer the given solution into 100mL standard flask and make the solution carefully. Pipette out 20mL of the made-up chloride solution into a clean conical flask. Add 1mL of 2% K₂CrO₄ indicator solution (The solution turns yellow in color). Titrate it against the standard AgNO₃ solution taken in the burette. Shake the conical flask continuously and continue to titrate until the reddish-brown tinge of the solution does not disappear. Note down the burette reading and repeat for concordant values.

TABLE I: Titration I- Standardization of Silver Nitrate solution

S.No	Vol. of std. NaCl	Burette	reading	Vol. of	Indicator
	soln (mL)	(mL)		AgNO ₃	
		Initial	Final	(mL)	
1					K ₂ CrO ₄
2					
3					

Volume of AgNO₃ Solution (V₁)=...mL Normality

of AgNO₃ Solution $(N_1) = ?$

Volume of NaCl Solution (V_2) = 10 mL

Normality of NaCl Solution (N_2) = 0.05 N

$$N1 = (V_2 \times N_2) / V_1$$

Normality of AgNO₃ Solution $(N_1) = ----N$

TABLE II

Titration II- Estimation of amount of chloride in a water sample

S.No	Vol. of the sample soln	Burette	reading	Vol. of	Indicator
	(mL)	(m	L)	AgNO ₃ (mL)	
		Initial	Final		
1					K ₂ CrO ₄
2					
3					

Volume of Chloride solution $(V_1) = 10 \text{mL}$

Normality of Chloride solution $(N_1)=?$

Volume of AgNO₃ Solution $(V_2) = ...mL$

Normality of AgNO₃ Solution (N₂)= from titration I

$$N_1 = (V_2 \times N_2) / V_1$$

Normality of Chloride solution $(N_1) = N$

Therefore, the amount of chloride present in the whole of the given solution

= (Normality of the ch	loride solution x Equivalent weight of Chloride) /10
=(N*35.5)/10	
=gm/Lit	

Result:

Amount of chloride present in the whole of the given solution is $= \frac{\text{gm/Lit}}{\text{chloride}}$

Bottle number =....

Rubrics for Evaluation	Marks
Experiment, Observation, Calculation	
% of error	
Signature of the teacher with date	

6. Determination of molecular weight of a polymer by Viscosity average method

EXPERIMENT NO.

DATE:

AIM:

To determine the molecular weight of a polymer in solution by using a viscometer

Apparatus required:

Ostwald's viscometer, Stop watch, Standard flasks, Graduated pipette and Suction bulb

Reagents required:

Polymer (PVA) and water

Principle:

Measurement of solution viscosity offers a simple and convenient method for molecular weight determination if polymer is soluble in a suitable solvent. In a capillary viscometer (Ostwald /Ubbelhode), the viscosity of a liquid is proportional to the time taken by a known volume of liquid to flow through a capillary under a specified hydrostatic pressure at a fixed temperature. Using Poiseuille's equation it is possible to show that if t, η and r are the flow time, viscosity and density of a solution respectively; and t_0 , η_0 and r_0 are those of pure solvent, then,

$$\eta/\eta_{\theta} = r/r_{\theta} * t/t_{\theta}$$

The value of η/η_{θ} is known as the **relative viscosity** η_{rel} . In dilute solutions, which are often employed for molecular weight determination, r is not much different from r_{θ} and hence

$$\eta_{rel} = \eta/\eta_{\theta} = t/t_0$$

Specific viscosity η_{sp} is defined as

$$\eta_{sp} = \eta - \eta_{\theta} / \eta_{\theta} = \eta / \eta_{\theta} - 1$$

$$= \eta_{rel} - 1$$

Reduced viscosity η *red* is equal to the ratio of the relative viscosity to concentration of the sample,

$$\eta_{red} = \eta_{sp}/C$$

The Staudinger – Mark-Houwink equation which relates η_i with molecular weight

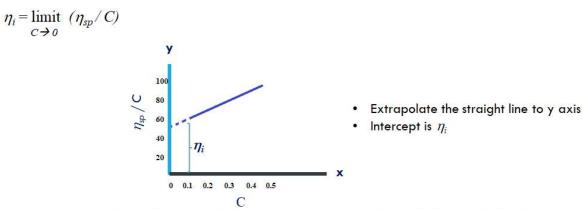
$$\eta_i = K(M)^a$$

For polyvinyl alcohol solution, $K = 45.3 \times 10^{-3}$ and a = 0.64.

where "M" is molecular weight of the polymer, η_i is intrinsic viscosity, "K" is an empirical parameter characteristic of a particular solute-solvent pair and 'a' is a shape parameter. From known values of K and a, molecular weight can be determined.

Intrinsic viscosity η_{i}

A plot of η_{sp}/C (reduced viscosity) vs C is a straight line for dilute solutions, the intercept



A plot of η_{sp} / C (reduced viscosity) vs C of a polymer solution to find out intrinsic viscosity

$$\eta_i = K(M)^{\alpha}$$

Procedure:

Preparation of various concentrations of polymer in water (solvent).

1 % of polymer solution in water will be supplied. We need to prepare at least 5 dilutions viz., 0.1, 0.2, 0.3, 0.4 and 0.5 % polymer in water before carrying out the experiment.

Dilutions can be done by using volumetric expressions:

$$V_1N_1 = V_2N_2$$

Eg. To prepare 25 mL of 0.2 % diluted solution from a 1% solution, volume is

$$V_1 = V_2 N_2 / N_1 = 25 \text{ mL x } 0.2 \% / 1\% = 5 \text{ mL}$$

Similarly, any other dilutions can be prepared by the above method.

S. No	Concentration of the polymer solution	Time of flow in sec (average)	Relative viscosity $\eta/\eta_0 = t/t_0$	Specific Viscosity $\eta_{sp} = \eta / \eta_0 - 1$	Reduced viscosity $(\eta_{red}) = \eta_{sp}/C \times 100$
1	Pure solvent	t_0			
2	0.1 %	$t_{\rm s}$			
3	0.2 %	$t_{\rm s}$			
4	0.3 %	$t_{\rm s}$			
5	0.4 %	$t_{\rm s}$			
6	0.5 %	$t_{\rm s}$			

Calculation:

$$\begin{split} \eta_i &= K \left(M \right)^{\alpha} \\ &\log \eta_i = \log K + \alpha \log M \\ &\alpha \log M = \log \eta_i - \log K \\ &\log M = \frac{\log \eta_i - \log K}{\alpha} \\ M &= \text{Antilog } \frac{[\log \eta_i - \log K]}{\alpha} \end{split}$$

Result:

Volume of polymer solution used for each measurement: mL

The average molecular weight of the given polymer is g/mol.

Rubrics for Evaluation	Marks
Experiment, Observation, Calculation and	
Graph	
% of error	
Signature of the teacher with date	

7. Determination of the strength of a mixture of Acetic Acid and Hydrochloric Acid by Conductometry

EXPERIMENT NO. DATE:

Aim:

To estimate the strength of mixture of acetic and hydrochloric acid present in a given mixture using conductometric titration.

Apparatus and reagents required:

Conductivity meter, Conductivity cell (Platinum plates), Burette, Pipette, Conical flask, standard measuring flask (SMF), NaOH, given unknown solution.

Principle:

The principle of conductometric titration is based on the fact that during the titration, one of the ions is replaced by the other ions and invariably these two ions differ in the ionic conductivity with the result that conductivity of the solution varies during the course of titration. During the titration of strong base against a strong acid is carried out, there are two break points observed in the graph. The first break point corresponds to the neutralization of strong acid (Eqn.1). After the complete neutralization of strong acid, at the second break point the neutralization of weak acid starts and conductivity increases slightly as unionized weak acid becomes the ionized salt (Eqn.2). Finally, the conductance increases due to the excess of strong base OH⁻ ions as the titrant.

$$NaOH + HCl \rightarrow NaCl + H_2O....$$
 (Eqn.1)

When the whole strong acid is consumed, base reacts with weak acid and

$$NaOH + CH_3COOH \rightarrow CH_3COO^- + Na^+ + H_2O \dots (Eqn.2)$$

Procedure:

Make up the given mixture of acid to 100 mL using distilled water in standard measuring flask. Pipette out 10 mL from this made-up solution into a clean beaker and then diluted to ~ 100 mL using distilled water. Immerse conductivity cell into the beaker and measure conductance for

every 0.5mL addition of NaOH from the burette and stir the solution for each addition. After complete neutralization, the amount of acid present in the given mixture is determined based on the volume of base consumed. Volume of base consumed for strong acid and weak acid are determined by plotting a graph between conductance (Y axis) and volume of NaOH added (X axis), where first end point corresponds to strong acid and the second end point corresponds to weak acid.

Model graph:

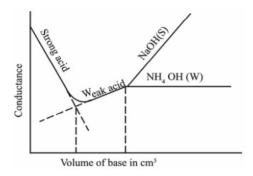


Table: Titration of Mixture of Acids Vs Base

S. No	Volume of NaOH added (mL)	Conductance (ohm ⁻¹)
1	0	
2	0.5	
3	1	
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		

14	
15	

Calculation:

I Determination of	f strength	of HCl:
--------------------	------------	---------

Volume of mixture $= V_1$ (20mL)

Normality of HCl solution $=N_1$

Volume of NaOH = V_2 (E_1 from the graph)

 $= N_2 (0.1N)$ Normality of NaOH

$$V_1N_1 = V_2N_2$$

$$N_1 = V_2 N_2 / V_1$$

The strength of the given HCl solution = ____N

II Determination of strength of Acetic acid:

 $=V_1$ (20mL) Volume of mixture

Normality of acetic acid $=N_1$

= V_2 (E_2 - E_1 from the graph) Volume of NaOH

Normality of NaOH $= N_2 (0.1N)$

$$V_1N_1=V_2N_2\\$$

$$N_1 = V_2 N_2 / V_1$$

The strength of the given acetic acid = _____N **Result:**

- The strength of HCl present in the whole of the given solution ______ N.
 The strength of CH₃COOH present in the whole of the given solution ______ N.

Bottle number =----

Rubrics for Evaluation	Marks
Experiment, Observation, Calculation and	
Graph	
% of error	

Signature of the teacher with date	

8. Determination of strength of an acid using pH meter

EXPERIMENT NO.

DATE:

Aim

To find out the strength of given HCl by titrating it against NaOH (0.1N) using pH meter.

Apparatus Required

250 and 100mL beaker, glass rod, 10mL pipette, 100mL standard measuring flask, pH meter,

Reagents Required

NaOH solution (0.1N), HCl sample solution, distilled water.

Principle

A pH meter is a scientific instrument that measures the difference in electrical potential between a pH electrode and the reference electrode with respect to hydrogen ion concentration which is expressed as "pH value". Initially there is a little change in pH due to the change in electrode potential that depends upon the fraction of H⁺ ions removed by a base NaOH when added in small increments to the acid (HCl). As the end point is reached, the fraction of H⁺ ions removal increases rapidly by the base that causes a rapid change in pH of the test solution.

$$H^{+} + Cl^{-} + Na^{+} + OH^{-} \rightarrow Na^{+} + Cl^{-} + H_{2}O$$

Procedure

Make up the given HCl solution to 100mL using distilled water. Pipette out 10mL of this made-up solution into a clean 250mL beaker and add 100mL of distilled water. Dip the pH meter into the solution and note the pH. Fill the burette with standard NaOH (0.1N) solution and add 1mL increments into the beaker, and measure the pH after each addition. Continue the process till and well beyond the neutralization point as indicated by an abrupt change in pH. Calculate the range at which sudden rise in pH occurs. Perform the similar titration by adding 0.2mL increments of standard NaOH close to the end point (1mL on either side of the range). Tabulate the pH measured corresponding to each addition.

Plot 2 graphs by taking

- 1. pH vs Volume of NaOH added
- 2. Δ pH/ Δ V vs Volume of NaOH added.

The exact endpoint should be calculated from the second graph.

Model Graphs

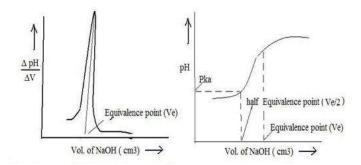


Table 1 Pilot Titration (Standard NaOH Vs Unknown HCl)

S.No	Volume of NaOH added (mL)	pН

Table 2 Fair Titration (Standard NaOH Vs Unknown HCl)

S.No	Volume of NaOH added (mL)	pН	ΔрН	ΔV	ΔρΗ/ΔV

Calculation

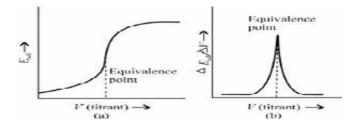
Volume of HCl	$= V_1 mL (10 mL)$
Normality of HCl	$= N_1$
Volume of NaOH	= V2mL (from the 2 nd graph)
Normality of NaOH	= N2 (0.1N)
$V_1N_I =$	V_2N_2
$N_1 = V_2 N_1$	$_{2}$ / V_{1}
The strength of given I	HCl solution =N.
Result	
The strength of given I	HCl solution is =N
Bottle number =	

Rubrics for	Evaluation			Marks
Experiment,	Observation,	Calculation	and	
Graphs				

% of error	
Signature of the teacher with date	

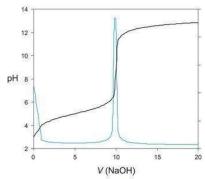
V Sample Questions

- 1. What is permanent and temporary hardness? Give salts responsible for permanent and temporary hardness in water.
- Calculate permanent hardness for sample hard water if the volume of the boiled water sample pipetted is 100mL and volume of EDTA consumed is 13.4mL. The volume EDTA consumed for standard hard water sample is 19.6mL.
- 3. What type of reaction does silver nitrate and sodium chloride undergo?
- 4. Calculate the normality of AgNO₃ when 12.5 mL of it is consumed for 10 mL of NaCl solution?
- 5. What are the ions responsible for alkalinity in water sample?
- 6. Give the type of reaction that is involved in the determination of amount of mixture of bases by titration?
- 7. Calculate the amount of Na₂CO₃ when the Phenolpthalein end point is 23.5 mL and Methyl orange end point is 46.9mL for 50 mL of made-up solution containing the mixture [pipette solution] when the Normality of acid is 0.0478N.
- 8. What happens to conductance when a solution is diluted?
- 9. When acid is titrated against base by conductometric method, the Conductance decreases and then increases. Why?
- 10. Calculate the Normality of acid when the intersection point from the graph meets at 8.9mL in x-axis for 10 mL of acid solution with 0.1N solution of base.
- 11. What is the oxidizing agent used in the potentiometric titration?
- 12. What is the indicator and reference electrode used in the potentiometric titration?
- 13. Calculate the amount of Fe²⁺ ions present in whole of the given solution if volume of pipette solution 10mL. Let us assume that the peak point in the following 2nd graph meet at 9.5mL for Potentiometric titration. Let the normality of the standard solution be 0.09N.



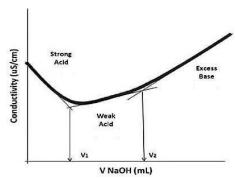
- 14. Give the structure for Polyvinyl alcohol and Polystyrene.
- 15. What is the solvent used for the dissolution of PVA and Why?

- 16. Intrinsic viscosity from the graph meeting at y-axis after extrapolating is 66 for the given polymer. Calculate Molecular weight when the k and α for PVA is 45.3 * 10⁻³ and 0.64 respectively.
- 17. What is the relation between pH and EMF of the cell for acid base titration? Give the model graphs for the pilot and fair titrations. Find the peak point for the above graph and also calculate the



Normality of the acid if the normality of the base is 0.11N and pipette solution amount is 10mL.

- 18. What are weak and strong acids? Give examples.
 - a. $CH_3COOH + Na + OH \rightarrow ?$
 - b. $H + + Cl + Na + + OH \rightarrow$?
- 19. Give reasons for the variation of curves in the graph given below. Let V1 be [from the graph] 5.8 mL and V2 be 10.7mL. Calculate the Normality of strong acid and weak acid if the N of the base be 0.6N



and the pipette solution be 20 mL.

N.B: The amount of Pipette solution, Normality is changed for few experiments.