



UNIVERSITY OF PETROLEUM & ENERGY STUDIES

PRACTICAL MANUAL

**CHEMISTRY LAB
(CHEM 1101)**

B.Tech (Sem. I & II)

(All Branches)

**DEPARTMENT OF CHEMISTRY
August 2017**

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EXPERIMENT-01

OBJECT:

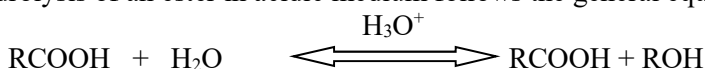
To determine the rate constant and order of the reaction of the hydrolysis of an ester (methyl / ethyl acetate) at 25⁰ C in the presence of hydrochloric acid.

REQUIREMENT:

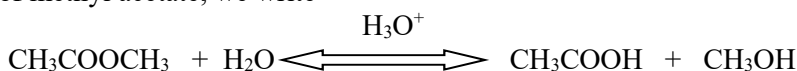
Pipette, conical flask, ice, stopwatch, burette, water bath, methyl acetate, HCl, NaOH, phenolphthalein indicator.

THEORY:

The hydrolysis of an ester in acidic medium follows the general equation:



In case of methyl acetate, we write



Since water is to be taken in large excess, its concentration remains practically constant during the hydrolysis. The rate equation for the above reaction has been found to be:

$$R = K [\text{CH}_3\text{COOCH}_3]$$

It is a first order reaction but its molecularity is 2, and it is therefore, is called pseudo unimolecular reaction. Since hydrolysis produces acetic acid, the rate of the reaction can be studied by titrating the acid formed against a standard solution of an alkali, say NaOH. The reaction can be stopped at any instance by lowering temperature of the reaction zone, using the ice. Its rate constant, K is calculated using integrated rate equation of the first order

$$K = \frac{2.303}{t} \log_{10} \frac{V_{\infty} - v_0}{V_{\infty} - v_t}$$

V_0 = Titration reading in the beginning

V_t = Titration reading after time 't'

V_{∞} = Titration reading after infinite time

PROCEDURE:

- (i) Now fill the burette with 0.1 N NaOH and keep phenolphthalein indicator nearby for ready use.
- (ii) In a beaker take 5mL ester and add 50mL of HCl in it.
- (iii) Start the stop watch. Pipette 5 ml of the reaction mixture and transfer into a flask containing ice cold water. This will stop the reaction due to low temperature. Add few drops of phenolphthalein in it and titrate it quickly against NaOH solution. Note the end point (Shown by appearance of pink colour). This reading is called V_0 as the time is zero.
- (iv) Now when the stop watch shows 10 minutes, take out again 5 ml from the reaction mixture to another ice cooled conical flask and titrate again quickly. Note the burette reading. It is V_t as $t=10$ minutes. Now this is to be repeated after 20, 30 and 40 minutes. These readings will give the values of V_t at different intervals. Now heat the 10-12mL of stock solution to ~70⁰ C for about half an hour. This will heat the reaction mixture to complete the hydrolysis. Take

5ml of it and titrate this also against the NaOH solution. This reading is V_{∞} . Heating has reduced the time for completion of the reaction.

(v) Do not refill the burette. Continue the reading till last one.

(vi) Plot graph between $\log_{10} \frac{V_{\infty} - V_0}{V_{\infty} - V_t}$ (y axis) and time (x-axis), and calculate K by using

$$K = \text{Slope} \times 2.303.$$

OBSERVATIONS & CALCULATIONS:

Time in mins	Volume of NaOH used in titration (ml)	$(V_{\infty} - V_t)$ ml	K (time^{-1})
0	V_0	$V_{\infty} - V_0$	
10	V_1	$V_{\infty} - V_1$	K_1
20	V_2	$V_{\infty} - V_2$	K_2
30	V_3	$V_{\infty} - V_3$	K_3
40	V_4	$V_{\infty} - V_4$	K_4
∞	V_{∞}		

RESULT:

i) Order of the reaction

ii) Rate constant (From Graph)

PRECAUTIONS:

i) All the glass apparatus must be clean.

ii) All solution must be prepared in the deionized water.

iii) Temperature of the bath must not vary beyond $\pm 0.5^{\circ} \text{C}$

iv) The reaction must be immediately arrested by adding ice cold water in the part of reaction mixture taken out.

EXPERIMENT-02

OBJECT:

To determine the moisture, volatile and ash contents in a given coal sample by proximate analysis.

REQUIREMENTS:

Powdered coal, analytical balance, electric oven, desiccator, silica crucible, tong, Muffle furnace

THEORY:

Proximate analysis is a type of assay used in the determination of different constituents present in the coal sample.

- 1. Moisture:** In coal sample, moisture may be of two types:
 - (i) Free or surface moisture, which is lost on air drying.
 - (ii) Internal moisture, which is retained by air dried coal.
- 2. Volatile matter:** It consists of a complex mixture of gaseous and liquid products resulting from the thermal decomposition of coal substances.
- 3. Ash content:** It is the non-combustible content left after burning of organic matter from the coal.
It may again be of two types:
 - (i) Fixed or inherent ash, intimately dispersed within the mass of the coal.
 - (ii) Free or extraneous ash, occurring in different layers of coal.

PROCEDURE:

- 1. Determination of moisture:** 1g of coal sample is taken in the pre-weighed silica crucible. It is kept inside the electric oven at 105 - 110°C for an hour. It is cooled in the desiccator and weighed. The difference in weight is reported in percentage as the amount of moisture in coal.
- 2. Volatile matter:** The dried coal sample after determining moisture content is heated in a muffle furnace at about 925°C for exactly 7 min with a lid. It is cooled in the desiccator without lid and weighed. Loss in weight gives the percentage of volatile matter.
- 3. Ash:** The residual coal in the crucible after the determination of volatile matter, is heated without lid in muffle furnace at about 725°C for half an hour for constant weight. Again it is cooled in desiccator. The amount of unburnt carbon residue in crucible is ash which can be reported in percentage.

OBSERVATIONS AND CALCULATIONS:

1. Moisture:

Weight of empty crucible = w_1 g

Weight of crucible + sample = w_2 g

Weight of sample = $(w_2 - w_1)$ g

Weight of (crucible + sample) after heating at 110°C = w_3 g

Moisture contents = $(w_2 - w_3)$ g

$$\% \text{ of moisture} = \frac{w_2 - w_3}{w_2 - w_1} \times 100$$

2. Volatile matter:

Weight of (crucible + sample) after heating at 925°C for 7 min = w_4 g

Volatile matter = $(w_3 - w_4)$ g

$$\% \text{ of volatile matter} = \frac{w_3 - w_4}{w_2 - w_1} \times 100$$

3.Ash:

Weight of (crucible + ash) = w_5 g

Ash contents = $(w_5 - w_1)$ g

$$\% \text{ of ash} = \frac{w_5 - w_1}{w_2 - w_1} \times 100$$

RESULT:

The given coal sample contains:

- (i) Moisture.....%
- (ii) Volatile matter.....%
- (iii) Ash.....%

PRECAUTIONS:

1. To avoid mechanical loss due to rapid removal of volatile matter, the temperature should be raised at a slow rate.
2. After or before weighing the crucible, it should not be held with fingers. The tong should be used for putting the crucible inside the oven / furnace.
3. The weighing should be made up to fourth decimal place.

EXPERIMENT-03

OBJECT:

To determine the strength of given HCl solution by titrating it against standard NaOH solution conductometrically.

APPARATUS REQUIRED:

Conductivity meter, conductivity cell, beaker (250 ml), burette (50 ml), volumetric flask (100 ml), glass rod.

CHEMICALS REQUIRED:

KCl solution (0.1 N), standard NaOH solution (0.5 N), unknown acid (HCl) solution, conductivity water.

THEORY:

Electrolytic conductivity is a measure of the ability of a solution to carry electric current. Conductometric titrations work on the principle of Ohm's law ($R = E/I$; $C = 1/R$). During ionic reactions, the conductance may either increase or decrease depending upon the nature of the electrolyte involved.

When hydrochloric acid solution (HCl) is titrated with sodium hydroxide solution (NaOH), the highly mobile hydrogen ions ($\lambda_{H^+}^\circ = 350 \text{ ohm}^{-1} \text{ cm}^{-1}$) are progressively replaced by slower moving sodium ions ($\lambda_{Na^+}^\circ = 50 \text{ ohm}^{-1} \text{ cm}^{-1}$) and the conductance of the solution decreases.



After the end point, the conductance of the solution rises sharply due to the presence of excess, highly mobile hydroxide ion ($\lambda_{OH^-}^\circ = 198 \text{ ohm}^{-1} \text{ cm}^{-1}$). Thus the neutralization of a strong acid by addition of a strong base leads to a minimum conductance at the end points. Therefore the nature of the plot (conductance of the solution versus volume of base added) will be as given below:

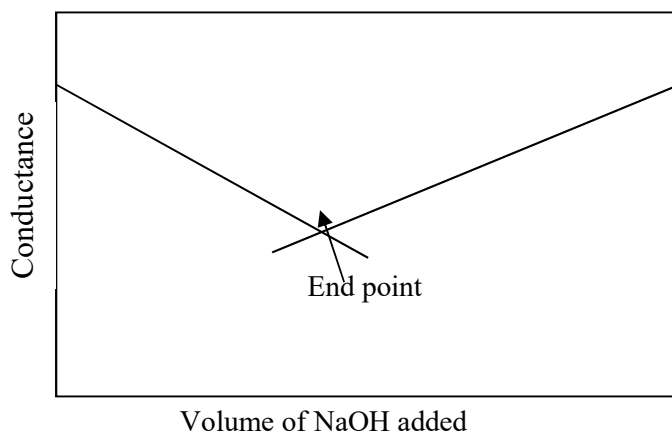


Fig. Conductometric titration of HCl vs NaOH

CALIBRATION OF CONDUCTOMETER:

Take the standard potassium chloride solution (14.11 mS) in a beaker. Immerse the electrode of conductivity meter in to the solution. Turn on the instrument and let it settle in the standard solution for few minutes. When the display gets stabilized, note down the value in mS. The difference in the obtained value and the conductance of standard KCl solution (14.11 mS) will be the instrumental error. Once calibration is completed, turn off the conductivity meter and wash the electrodes with distilled water.

PROCEDURE:

Fill the burette with 0.5 N NaOH solution. Take 50 ml of the given acid solution in a conductivity cell (250 ml beaker) with the help of volumetric flask. Dilute it with 50 ml conductivity water (double distilled water) so that the electrodes of the cell are completely dipped in the solution. Place the beaker below the burette. Immerse the electrode of conductivity meter into the solution and note the conductance of the solution. Now, add NaOH solution from burette in the acid solution, mix with glass rod and note the conductivity of the resulting solution after each addition of 0.5 ml of NaOH till almost a constant value of conductance is not obtained.

Plot the graph of conductance vs amount of NaOH added gradually.

OBSERVATIONS:

Volume of HCl used = 50 ml

Volume of total solution in conductivity cell = 100 ml

S.No.	Volume of NaOH added (ml)	Conductance of the solution (mS)	Conductance \pm error (mS)
1.	0.0		
2.	0.5		
3.	1.0		
4.	1.5		
5.	2.0		
6.	2.5		
7.	3.0		
8.	3.5		
9.	4.0		
10.	4.5		
11.	etc.		

CALCULATION:

Applying normality equation:

$$N_1 V_1 = N_2 V_2$$

Where, $N_1 \rightarrow$ Normality of acid

$V_1 \rightarrow$ Volume of acid used

$N_2 \rightarrow$ Normality of NaOH

$V_2 \rightarrow$ Volume of NaOH required to reach the end point.

$$\text{Strength of HCl} = \text{Normality} \times \text{Equivalent weight}$$

RESULT:

Strength of HCl solution = ----- g/lit.

PRECAUTIONS:

1. Stirring should be done after each addition of titrant.
2. All precautions regarding handling of the instrument should be observed.
3. Cell must properly be washed before using.

EXPERIMENT-04

OBJECT:

To determine the strength of given HCl solution by titrating it against standard NaOH solution pH-metrically.

APPARATUS REQUIRED: pH meter, electrode, beaker (100 ml), burette, pipette, glass rod.

CHEMICALS REQUIRED: Standard NaOH solution (0.5 N), unknown acid (HCl) solution, buffers of pH 4.0 and 9.2.

THEORY:

In order to express the acidity or alkalinity of a solution, one can use the hydrogen ion concentration. The pH of a solution is defined as negative logarithm of hydrogen ion concentration.

$$\text{pH} = -\log [\text{H}^+]$$

When an alkali is added to an acid solution, the pH of the solution increases slowly, which towards the approach of the equivalence point, the rate of change of pH of the solution is very rapid. From the sharp break in the curve, we can find the equivalence point, from which the strength can be calculated.

CALIBRATION OF pH METER:

Turn on the instrument, select the pH Mode and set the temperature control knob to 25°C. Rinse the electrode with deionized water and blot dry using a piece of tissue. Place the electrode in the solution of pH 4.0 buffer, allow the display to stabilize and then, set the display to read 4 by adjusting cal knob. After adjustment, remove the electrode from the buffer, rinse it with deionized water and blot dry using a piece of tissue. Now place the electrode in the solution of pH 9.2 buffer, allow the display to stabilize and then, note the display reading. The difference in display pH and 9.2 will be the instrumental error. Remove the electrode from the buffer, rinse it again with deionized water and blot dry using a piece of tissue.

PROCEDURE:

First standardize the pH-meter against a buffer of known pH. Now, first wash the glass electrode with distilled water and then with the given acid solution. Take 50 ml of given acid solution in a 100 ml beaker and add 50 ml of water so that the glass electrode is completely dipped in the solution. Note the pH of pure acid. Now add 1 ml of standard NaOH from the burette in the beaker, stir the contents well with glass rod and note the pH of the solution. Now keep on adding NaOH solution from the burette and note the pH of the solution after each addition of 1.0 ml of NaOH till almost a constant value of pH is not obtained.

OBSERVATIONS:

Volume of HCl used = 50 ml

Volume of total solution = 100 ml

S.No.	Volume of NaOH added (ml)	pH	pH \pm error
1.	0.0		
2.	1.0		
3.	2.0		
4.	3.0		
5.	4.0		
6.	5.0		
7.	6.0		
8.	7.0		
9.	8.0		
10.	9.0		
11.	etc.		

CALCULATIONS:

A curve is plotted in pH vs amount of alkali added. Find out the end point. Drop a straight line (parallel to x-axis) on the graph from pH 7. From the point of intersection, draw a perpendicular at x-axis. This will be the volume of NaOH (V_2) used for neutralization of acid.

Applying normality equation:

$$N_1 V_1 = N_2 V_2$$

(Acid) (Alkali)

$$\text{Strength of HCl} = \text{Normality} \times \text{Equivalent weight}$$

RESULT:

The strength of given acid solution isgm/l.

PRECAUTIONS:

1. The temperature knob of the pH-meter should be adjusted at room temperature.
2. After the addition of the alkali, the solution should be thoroughly stirred.
3. The pH-meter should be first standardized by taking a buffer of known pH.

EXPERIMENT-05

OBJECT:

To determine the amount of sulphate content in given samples by the gravimetric analysis.

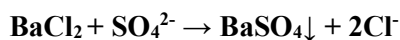
APPARATUS/REAGENT REQUIRED:

BaCl₂ solution, Unknown solution of Sulfur, Beaker, Silica crucible, Dessicator, Weighing Balance, HCl solution (6M).

THEORY:

Gravimetric analysis is a quantitative method for accurately determining the amount of a substance by selective precipitation of the substance from an aqueous solution. The precipitate is separated from the aqueous solution by filtration and is then weighed. Assuming that the chemical formula for the precipitate is known and that the precipitation reaction is stoichiometric (goes to completion) the mass of the substance/element in the original sample can be determined.

The solution of BaCl₂ reacts with SO₄²⁻ ions and precipitated in the form of BaSO₄ since solubility product BaSO₄ is very low (K_{sp} of BaSO₄ in water is 1.08 x 10⁻¹⁰ at 25°C)



PROCEDURE:

Take 25ml of unknown solution and add ~100 ml of distilled water. Heat the solution at 60-70°C for 10-15 minutes. Put a glass rod in the beaker.

1. Add hot Barium Chloride solution (5% w/v) so that BaSO₄ precipitate till complete precipitation is not achieved.
2. Now heat the content over hot plate for ~10 minute for complete coagulation of precipitates of BaSO₄.
3. Now, remove the beaker from hot plate and wait so that precipitates settle down. **Check that further precipitation of BaSO₄ is occurring or not by slight addition of BaCl₂ in the solution. If a further precipitation is occurring repeat the steps 1 & 2.**
4. Now filter the solution through Whatmann Ash less Filter paper number 40 or 42. Wash the precipitates with hot water to remove unwanted adsorbed components from the BaSO₄.
5. Transfer the filter paper along with precipitate in a pre-constantly weighed silica crucible and place in oven for drying at ~70-80 °C for 10 minutes.
6. Transfer the crucible in Muffle furnace carefully at heat the content for 30 minutes at 550 °C. Cool the crucible in a dessicator and weigh it carefully so that constant weight is obtained.

OBSERVATIONS:

Weight of empty crucible = x gm and Weight of crucible + precipitate = y gm

Therefore the weight of BaSO₄ precipitate will be = (y-x) gm

RESULT:

The given solution containsgm of BaSO₄.

PRECAUTIONS:

1. Precipitation should be carried out in hot and dilute solution.
2. The precipitated solution should be digested properly.
3. During digestion (i) Glass rod must be inside the beaker, (ii) beaker must be covered with beaker and (iii) there must be no bumping in the solution.
4. Filtration must strictly be done by decantation method and during ignition there must be no loss of precipitates.
5. Red-hot crucible must not be placed directly in the dessicator.

EXPERIMENT-06

OBJECT:

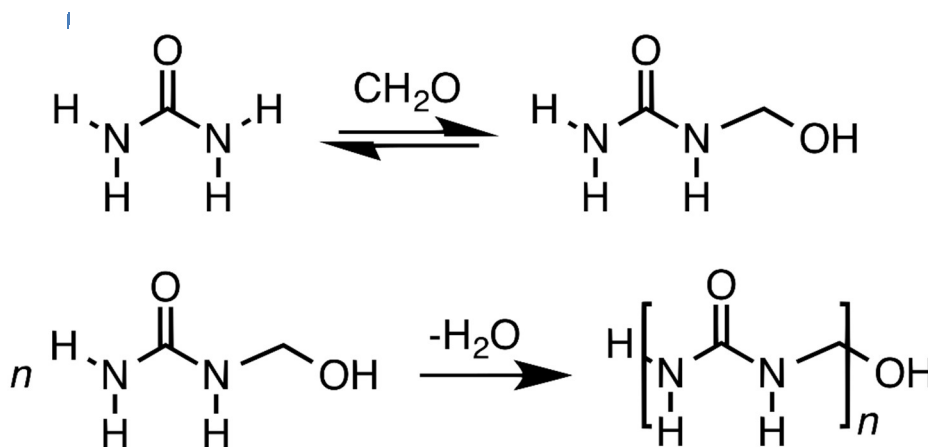
To prepare urea-formaldehyde (UF) resin.

APPARATUS/REAGENT REQUIRED:

Formaldehyde (40%), urea, conc. H_2SO_4 , beaker, glass funnel, filter paper.

THEORY:

Amino resins are the condensation products obtained by condensation reaction of urea with formaldehyde in acidic or alkaline medium. The first step is the formation of dimethyl urea



Urea-formaldehyde Resin

PROCEDURE:

Take 5ml of 40% formaldehyde solution in a 100ml beaker. Add about 2.5g of urea with stirring until a saturated solution is obtained. Add a few drops of conc. H_2SO_4 with constant stirring. Continue stirring till a white colored solid mass is not obtained. Wash the resin with water, dry and weigh it.

RESULT:

- (i) Physical appearance:.....
(ii) Yield:.....g

Note: Attach a small amount of sample in polybag in your practical copies.

PRECAUTIONS:

1. Reaction is exothermic and hence addition of conc. H_2SO_4 should be done very slowly.
2. The reaction mixture should be stirred continuously.

EXPERIMENT-07

OBJECT:

To determine the strength of given sodium chloride solution by titrating it against standard N/50 silver nitrate solution using potassium chromate as an internal indicator

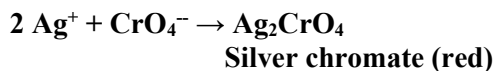
REAGENTS AND APPARATUS USED:

AgNO₃ solution (N/50), NaCl solution, potassium chromate indicator, burette, pipette, conical flask

THEORY:

When silver nitrate is added to a solution of sodium chloride containing a few drops of potassium chromate, white silver chloride is precipitated initially. As soon as all the chloride ions have been precipitated out, even a drop of silver nitrate added in excess, gives a red precipitate of silver chromate, indicating the end point.

The solubility product of silver chloride is lower than that of silver chromate. Hence, as long as the chloride ions are available, silver chloride is precipitated and as soon as the chloride ions have been precipitated out, even a slight excess of Ag⁺ ions produces red coloured insoluble silver chromate.



Procedure: Fill the burette with standard silver nitrate solution. Pipette out 10 ml of sodium chloride in a conical flask and add 2 to 3 drops of potassium chromate indicator and titrate it with silver nitrate slowly (appearance of white color indicates the formation of AgCl) and continue the titration until red precipitate appears. Note down the burette reading and continue the titration until concordant reading occurs.

Observation Table:

S. No.	Vol. of NaCl solution, ml	Reading of burette		Vol. of AgNO ₃ solution used , ml (Final – Initial)
		Initial	Final	
1				
2				
3				

Calculations: $N_1V_1 = N_2V_2$
(AgNO₃) (NaCl)

N_1 = Normality of AgNO₃ = 1/50

N_2 = Normality of NaCl

V_1 = volume of AgNO₃ (Concordant Reading)

V_2 = volume of NaCl = 10 ml

$$N_2 = N_1V_1 / V_2$$

Strength of sodium chloride = Normality (N_2) X Equivalent wt of NaCl g/lit

(Equivalent Weight of sodium chloride = 58.5)

Result : The strength of given NaCl solution is _____ g/lit

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

EXPERIMENT – 08

OBJECT:

To prepare N/30 ferrous ammonium sulphate solution and to determine the strength of given ferrous ammonium sulphate solution using potassium dichromate as intermediate solution and diphenyl amine as internal indicator.

REQUIREMENTS:

Ferrous ammonium sulphate (Mohr's salt), unknown ferrous ammonium sulphate solution, standard potassium dichromate solution, diphenyl amine, dilute sulphuric acid, burette, pipette, conical flask.

INDICATOR:

Diphenyl amine, $(C_6H_5)_2NH$.

END POINT:

Change of colourless, greenish solution to bluish-violet.

THEORY:

Potassium dichromate oxidizes ferrous sulphate present in Mohr's salt into ferric sulphate in the presence of dilute sulphuric acid.



Diphenyl amine, in the presence of potassium dichromate, is converted from benzenoid to quinonoid form which is of bluish-violet colour. As long as single ferrous ion is present in the reaction mixture, no change in colour of the solution is there but as all the ferrous ions have been converted to ferric ions, addition of single drop of potassium dichromate converts benzenoid form of diphenyl amine (colourless) to its quinonoid form (bluish-violet) marking the end point of the reaction.

PROCEDURE:

It completes in three stages:

1. Preparation of known (standard) ferrous ammonium sulphate solution:

Weigh out exactly 1.3066 g of Mohr's salt.

$$W = \frac{NVE}{1000} \text{ g}$$

Where, N → Normality of solution required (N/30)

V → Volume of solution to be prepared (100 ml)

E → Equivalent weight of ferrous ammonium sulphate (392)

Transfer it into a 100ml measuring flask and dissolve it in about 1ml of dilute sulphuric acid. Now make up the solution upto the mark with distilled water. This is N/30 ferrous ammonium sulphate solution.

2. Titration of known ferrous ammonium sulphate solution against standard potassium dichromate solution:

Pipette out 10ml of N/30 ferrous ammonium sulphate solution in a conical flask and add about 5ml of dilute sulphuric acid to it. Now add 4-5 drops of indicator to the reaction vessel and start titrating ferrous ammonium sulphate solution in a conical flask against standard potassium dichromate solution filled in the burette. Continue titration till the greenish solution of the reaction vessel does not change to bluish-violet. This is the end point of the reaction.

Repeat the same titration for concordant readings.

3. **Titration of unknown ferrous ammonium sulphate solution against standard potassium dichromate solution:** Repeat exactly the same titration with unknown (supplied, whose strength is to find out) ferrous ammonium sulphate solution and take concordant readings.

OBSERVATIONS:

Weight of empty watch glass = x g

Weight of watch glass + Mohr's salt = y g

Weight of Mohr's salt = (y – x) g

Titration of known ferrous ammonium sulphate (FAS) solution against standard K₂Cr₂O₇:

S. No.	Volume of FAS (ml)	Reading of burette		Volume of K ₂ Cr ₂ O ₇
		Initial	Final	
1.	10	0		
2.	10	0		
3.	10	0		

Let, volume of K₂Cr₂O₇ used with known FAS solution = V₁ ml

Titration of unknown ferrous ammonium sulphate (FAS) solution against standard K₂Cr₂O₇:

S. No.	Volume of FAS (ml)	Reading of burette		Volume of K ₂ Cr ₂ O ₇
		Initial	Final	
1.	10	0		
2.	10	0		
3.	10	0		

Let, volume of K₂Cr₂O₇ used with unknown FAS solution = V₂ ml

CALCULATIONS:

$$\text{Strength of given ferrous ammonium sulphate solution} = \frac{10 \cdot w \cdot V_2}{V_1} \text{ g/l}$$

RESULT:

The strength of given ferrous ammonium sulphate solution isg/l.

PRECAUTIONS:

1. While preparing known ferrous ammonium sulphate solution, never dissolve the salt directly in water.
2. Properly rinse the burette and pipette with the respective solutions.
3. Add the titrant from the burette drop wise with constant stirring.
4. After every titration, wash the conical flask well with water.