EXPERIMENT: 01

AIM: Preparation of Fluorescein.

REQUIREMENT: Phthalic anhydride, resorcinol, zinc chloride.

REACTION

**PROCESS:**
The 15gm of Phthalic anhydride and 22gm of resorcinol is heated in oil bath to 180°C in a 250 ml. round bottom flask. As soon as the temperature has reached to 180°C, 7 gm of fused ZnCl₂ is added gradually during 10 minutes, the melt being stirred mechanically. After the zinc chloride is added, the temperature is raised to 210°C and kept at this point until the mass become solid. The melt is broken out of the vessel with knife and dissolved in dilute caustic soda. After filtering, hydrochloric acid is added which precipitates the dye.

**CALCULATION:**

% yield = (Practical yield/ Theoretical yield) 100

**RESULT:**

1. Theoretical yield: *******gm.
2. Practical yield: *******gm.
3. Percentage yield: *******%
EXPERIMENT: 02

AIM: Preparation of Indophenol blue.

REQUIREMENT: Nitrosodimethyl aniline hydrochloride, zinc dust, α-naphthol, caustic soda, potassium dichromate.

REACTION:

\[
\text{H}_3\text{C} \quad \text{N} \quad \text{CH}_3 \\
\text{HCl} \\
\text{NO} \\
\text{Zn} \quad + \quad \text{K}_2\text{Cr}_2\text{O}_7^+ \\
\text{NaOH} \\
\text{OH} \\
\text{H}_3\text{C} \quad \text{N} \quad \text{CH}_3
\]

PROCESS: The 10gm of NDAH is dissolved in 100 ml of water and reduced by adding 10gm Zinc dust and warming solution to 45-50°C for ½ hour to 1 hour. The mixture becomes colorless & is filtered from the zinc and zinc chloride. The solution of aminodimethyl aniline is mixed with the naphthol solution prepared by dissolving the 12gm of naphthol in 2.3gm of caustic soda and a 50cc of water and the mixture is added to the solution of 10gm dichromate in 100cc of water. The mixture is well stirred mechanically and 30 to 40% acetic acid is slowly added until the acidic reaction is obtained. The dye is precipitated filtered and dry.

PROPERTIES: Indophenol blue is a dark brown powder, insoluble in water.

CALCULATION:

\[
\% \text{ yield} = \frac{\text{Practical yield}}{\text{Theoretical yield}} \times 100
\]

RESULT:

1. Theoretical yield: \[\text{---gm.}\]
2. Practical yield: \[\text{---gm.}\]
3. Percentage yield: \[\text{---}\%\]
EXPERIMENT: 03

AIM: Preparation of Mordant Yellow.

REQUIREMENT: m-Nitroaniline, Salicylic acid, Conc. hydrochloric acid, Sodium nitrite.

REACTION:

PROCESS: A mixture of 7gm of m-nitroaniline, 20ml of conc HCl and 40ml water is prepared in a 500ml beaker. This is heated until solution is completely clear. Solution is cooled to room temperature and then to 5°C with adding 40gm ice prepares a solution of 3.5gm NaNO₂ in 10ml water in a 100ml beaker. Add sodium nitrite solution into above prepared solution slowly keeping temperature below 5°C, prepare a solution of 7gm, salicylic acid in 30ml 20% Na₂CO₃. Cool to 5°C pour diazo solution in salicylic acid at 5°C. Make the bath alkaline by adding 20% Na₂CO₃ solution stir the reaction mixture at 5°C for 2 hours. Then add 10% NaCl solution at 45-50°C till salt dissolved completely, cool and stir at room temperature for one hour, till complete precipitation of dye. Filter and dry.

CALCULATION:

% yield = (Practical yield/ Theoretical yield) 100

RESULT:

1. Theoretical yield: *******gm.
2. Practical yield: *******gm.
3. Percentage yield: *******%
EXPERIMENT: 04

AIM: Preparation of Orange-I

REQUIREMENT: Sulphanilic acid, α-Naphthol, Sodium carbonate, Sodium nitrite, Conc. HCl solution.

REACTION:

\[
\begin{align*}
\text{SO}_3\text{H} & \quad \text{NaNO}_2 \quad \text{HCl} \\
\text{NH}_2 & \quad \text{N}_2\text{Cl} \\
\end{align*}
\]

\[
\begin{align*}
\text{SO}_3\text{H} & \quad \text{OH} \\
\end{align*}
\]

\[
\begin{align*}
\text{HO}_3\text{S} & \quad \text{N} \equiv \text{N} \\
\end{align*}
\]

PROCESS: Take 2.6gm of Sulphanilic acid, 0.7gm of Na\textsubscript{2}CO\textsubscript{3} and 25 ml water in 250 ml beaker. Warm until clear solution is obtained. Cool the solution to 5°C. Now prepare a solution of 1 gm sodium nitrite in 10 ml water in a 100 ml beaker. Add this solution to above prepared solution in a 150 ml beaker, take 2.6 ml conc. HCl and 15gm of ice. Add above prepared solution from beaker very slowly with stirring in 250 ml beaker. Dissolve 1.8gm of α -naphthol in 10 ml of cold 10% NaOH in a 500 ml beaker with 25 ml water. Cool to 5°C. In add above prepared solution with stirring keeping temperature 5°C. Dyestuff separates as crystalline paste. After 10minutes, heat the mixture until all solid dissolve. Add 5gm of NaCl and warm until this dissolves. Allow the solution to cool for ½ hr and then and in ice bath until crystallization complete. Filter and dry.

CALCULATION:

% yield = \((\text{Practical yield} / \text{Theoretical yield}) \times 100\)

RESULT:

1. Theoretical yield: \(\ldots\)gm.
2. Practical yield: \(\ldots\)gm.
3. Percentage yield: \(\ldots\)%
EXPERIMENT: 05

AIM: Preparation of Chrysodine dye.

REQUIREMENT: Aniline, Sodium nitrite, HCl, \textit{m}-phenylenediamine (MPDA).

REACTION:

\[ \text{NH}_2 \quad \xrightarrow{\text{NaNO}_2, \text{HCl}} \quad \text{N}_2 \text{Cl} \quad \xrightarrow{\text{NH}_2} \quad \text{N} = \text{N} \quad \text{H}_2\text{N} \quad \xrightarrow{\Theta} \quad \text{H}_2\text{N} \]

PROCESS: Prepare a solution of 9.3gm Aniline and 30cc of conc. HCl in a 500ml beaker. Now prepare a solution of 7.2gm sodium nitrite in 20cc of water. Allow the sodium nitrite in solution slowly to flow in the aniline solution keeping solutions temperature 5\(^\circ\)C. Now prepare a solution of 11gm \textit{m}-phenylenediamine in 50cc of cold water and 10cc of conc HCl the diazo solution is poured into the above solution keeping the temperature 5\(^\circ\)C. This addition will take about an hr stirred the mixture for \(\frac{1}{2}\) hour. The solution is now heated to 60\(^\circ\)C and 40cc of a saturated solution of salt is added. Cool to room temperature and then in to ice bath. Filter and dry.

PROPERTIES: Reddish brown crystals soluble in water with a yellow colour. Dyed wool, silk and tannin mordanted cotton orange.

CALCULATION:

% yield = \(\frac{\text{Practical yield}}{\text{Theoretical yield}}\) \times 100

RESULT:

3. Percentage yield: \_________%
EXPERIMENT: 06

AIM: Preparation of Naphthol Blue Black-6B dye.

REQUIREMENT: p-Nitroaniline, Sodium nitrite, H-acid, Aniline, HCl etc.

REACTION:

\[
\begin{align*}
\text{NH}_2 & \quad \text{OH} & \quad \text{NH}_2 \\
\text{NO}_2 & \quad \text{N}_2\text{Cl}^- & \quad \text{HO}_3\text{S} & \quad \text{SO}_3\text{H} \\
\text{NO}_2 & \quad \text{N}_2\text{Cl}^- & \quad \text{HO}_3\text{S} & \quad \text{SO}_3\text{H}
\end{align*}
\]


PROCESS:

(A) Dissolve 3.5gm of p-Nitroaniline by boiling with 10cc conc. HCl and 20cc water in 250 ml beaker cool it to room temperature and add 50gm ice to cool to 5°C. Then add solution of 3.5gm NaNO\textsubscript{2} in 10cc water with stirring at 5°C.

(B) Dissolve 8.5gm H-acid in 50cc water containing 10 gm Na\textsubscript{2}CO\textsubscript{3} in 500ml beaker. Neutralize any excess of alkali with HCl acid. Add slowly (A) into (B) with mechanical stirring and continue stirring for ½ hour. Then add to it solution of 2.3ml Aniline, 7.5ml conc. HCl and 1.8gm NaNO\textsubscript{2} prepared at 5°C stir for 1 hour. Then heat to 80°C and add 10 gm NaCl. Filter and dry.

CALCULATION:

\[
\text{% yield} = \left( \frac{\text{Practical yield}}{\text{Theoretical yield}} \right) 100
\]

RESULT:

3. Percentage yield: \________%
AIM: Preparation of Lake Red-D dye.

REQUIREMENT: Anthranilic acid, β-naphthol, NaCl, Sodium nitrite, HCl acid.

REACTION:

\[
\text{CONC. HCl} + \text{NaNO}_2 + \text{NaCl} \rightarrow \text{Lake Red-D dye}
\]

PROCESS: Take 2.6gm anthranilic acid, 0.7gm Na\textsubscript{2}CO\textsubscript{3} and 25ml. water in 250 ml beaker, warm until clear solution is obtained. Cool the solution to 5\(^\circ\)C. Now add a solution of 1.09 gm NaNO\textsubscript{2} in 10ml. of water. Now prepare a solution of 3.0ml conc. HCl and 15gm ice with 10ml water in 100ml. beaker. Add above prepared solution from beaker very slowly with stirring in beaker. Dissolve 1.8gm of β-naphthol in 10ml 10% NaOH in a 500ml beaker cool to 5\(^\circ\)C. In this solution, add above prepared solution with stirring. Dyestuff separates out as a crystalline paste. After 10 minutes, heat the mixture until all solid is dissolve. Add 5gm NaCl and warm. Allow the solution to cool for ½ hour and then in ice bath until crystallization complete. Filter and dry.

CALCULATION:

\[
\text{% yield} = \left( \frac{\text{Practical yield}}{\text{Theoretical yield}} \right) \times 100
\]

RESULT:

1. Theoretical yield: \(\text{-------gm.}\)
2. Practical yield: \(\text{-------gm.}\)
3. Percentage yield: \(\text{-------%}\)
EXPERIMENT: 08

AIM: Preparation of Fast Green-O. (Di-nitroso resorcinol)

REQUIREMENTS: Resorcinol, Sodium chloride, sodium nitrite, HCl acid etc.

REACTION:

\[
\begin{align*}
\text{Resorcinol} & \xrightarrow{HCl, NaNO}_2 \\
& \quad 0-5^\circ C
\end{align*}
\]

PROCESS: Dissolve 10gm resorcinol in 100ml of water. Add 20cc HCl and 25gm NaCl in above solution. Add ice and bring temperature to 0°C. Stir the solution mechanically. Prepare a solution of 13gm sodium nitrite in 50ml water. Allow nitrate solution to flow very slowly. This takes about 60 minutes. When liquid show acidic nature, allow stand for one hour. Filter the precipitate, wash with cold water and dry.

CALCULATION:

\[
\% \text{ yield} = \left( \frac{\text{Practical yield}}{\text{Theoretical yield}} \right) \times 100
\]

RESULT:

3. Percentage yield: \(-\)%
4. M.P.: \(-\)^\circ C
EXPERIMENT: 09

AIM: Preparation of Methyl Orange

REQUIREMENTS: Sulphanilic acid, sodium carbonate, sodium nitrite, N,N-Dimethylaniline, HCl, sodium chloride etc.

REACTION:

\[
\begin{align*}
\text{OH} & \quad \text{HCl} & \quad \text{N}_2\text{Cl}^- \\
\text{SO}_3\text{H} & \quad \text{NaNO}_2 & \quad 0-5^\circ\text{C} \\
\end{align*}
\]

\[
\begin{align*}
\text{H}_3\text{C}-\text{N-CH}_3 & \quad \text{HO}_3\text{S} & \quad \text{N=}=\text{N} \\
\text{SO}_3\text{H} & \quad \text{H}^+ & \quad \text{N}\quad\text{CH}_3
\end{align*}
\]

PROCESS:

(A) Dissolve 10gm Sulphanilic acid in the solution of 2.5gm Na\textsubscript{2}CO\textsubscript{3} in 100ml water. Add solution of 3gm NaNO\textsubscript{2} in 20ml of water. Cool the mixture in ice bath to about 5\degree C. Add a solution of 5ml HCl in 10 ml of water gradually keeping the temperature below 5\degree C.

(B) Prepare a solution of 6 ml N-N-Dimethylaniline in a mixture of 6ml conc. HCl and 20 ml of water. Cool to about 5\degree C. Add solution (B) into (A) with stirring. Make the whole bulk alkaline by addition 10% NaOH solution (20ml) drop wise. Then add 10ml saturated sodium chloride solution. Heat the mixture to boiling. Cool first to room temperature and then in ice bath. Filter and wash with cold water and dry. Recrystallized from hot water gives pale yellow crystal.

CALCULATION: % yield = (Practical yield / Theoretical yield) 100

RESULT:

1. Theoretical yield: \text{--------\,gm.}
2. Practical yield: \text{--------\,gm.}
3. Percentage yield: \text{--------\%}
EXPERIMENT: 10

AIM: Preparation of Disperse Dye.

REQUIREMENTS: \( p \)-Nitroaniline, Sodium nitrite, sodium carbonate, \( N,N \)-Dimethyl aniline conc HCl etc.

REACTION:

\[
\begin{align*}
\text{OH} & \quad \text{HCl} \\
\text{SO}_3\text{H} & \quad \text{NaNO}_2 \\
0-5^\circ\text{C} & \quad 0-5^\circ\text{C} \\
\text{N}_2^+\text{Cl}^- & \quad \text{H}_3\text{C}^-\text{N}^-\text{CH}_3 \\
\text{O}_2\text{N} & \quad \text{N}=\text{N} \\
\text{N} & \quad \text{N} \\
\text{CH}_3 & \quad \text{CH}_3
\end{align*}
\]

PROCESS: A mixture of 3.5gm \( p \)-Nitroaniline, 8ml of water and 10ml of conc. HCl acid is heated until solution is completely clear. Cool it to room temperature. Then add ice to bring the temperature at 5\(^\circ\)C. Add solution of 1.75gm sodium nitrite in 20ml water slowly. Keep the solution in ice bath at 0-5\(^\circ\)C. Then pour diazo solution at 0-5\(^\circ\)C in \( N,N \)-Dimethylaniline solution in 25 ml water and 5 ml conc. HCl acid. Add 10% 100 ml NaCl solution. Heat the mixture to 40-50\(^\circ\)C for 15 minutes. Filter and dry.

CALCULATION:

\[
\% \text{ yield} = \left( \frac{\text{Practical yield}}{\text{Theoretical yield}} \right) 100
\]

RESULT:

1. Theoretical yield: \( \_\_\_\_ \)gm.
2. Practical yield: \( \_\_\_\_ \)gm.
3. Percentage yield: \( \_\_\_\_ \)%
EXPERIMENT: 11

AIM: Preparation of Fast Red-A. (Rosaniline)

REQUIREMENT: Sodium naphthionate, sodium nitrite, β-naphthol, sodium hydroxide etc.

REACTION:

\[
\begin{align*}
\text{Na}_2\text{C}_{10}\text{H}_7\text{Na} & \xrightarrow{\text{HCl}} \text{N}_{\text{a}}\text{N}_2\text{C}_{10}\text{H}_7\text{Na} \\
\text{NaNO}_2 & \xrightarrow{0-5^\circ\text{C}} \text{N}_{\text{a}}\text{N}_2\text{C}_{10}\text{H}_7\text{Na} \\
\text{OH}^- & \xrightarrow{\text{Na}_2\text{C}_{10}\text{H}_7\text{Na}} \text{N}_{\text{a}}\text{N}_2\text{C}_{10}\text{H}_7\text{Na}
\end{align*}
\]

PROCESS: Dissolve 12gm of sodium naphthionate in 150cc of water. Add 10cc conc HCl acid. And cool it to 5°C. Then add 3.5gm NaNO₂ in 20ml. of water drop wise with constant stirring keeping the temperature 0-5°C. Prepare a solution of 7.5gm β-naphthol, 2.25gm sodium hydroxide in 25cc of water. Heat the mixture until it is completely clear. Add diazotized solution to the above prepared solution slowly, keeping temperature 5°C after an hours stirring, heat the mixture to 80°C for 15 minutes. Add 50 ml. of 10% NaCl solution. Cool to room temperature. Filter and dry.

CALCULATION:

\[
\% \text{ yield} = \frac{\text{Practical yield}}{\text{Theoretical yield}} \times 100
\]

RESULT:

1. Theoretical yield: \_\_\_\_\_\_\_gm.
2. Practical yield: \_\_\_\_\_\_\_gm.
3. Percentage yield: \_\_\_\_\_\_\_%
EXPERIMENT: 12

AIM: Preparation of p-Nitroso N,N-dimethyl aniline Hydrochloride.

REQUIREMENTS: N-N-Dimethylaniline, Hydrochloric acid, Sodium nitrite etc.

REACTION

\[
\begin{align*}
\text{H}_3\text{C} & \quad \text{N} \quad \text{CH}_3 \\
\text{C}_6\text{H}_4 & \quad \rightarrow \\
\text{H}_3\text{C} & \quad \text{N} \quad \text{CH}_3 \cdot \text{HCl}
\end{align*}
\]

\[
\text{HCl} \\
\text{NaNO}_2 \\
0-5^\circ\text{C}
\]

PROCESS: Dissolve 25ml. of N,N-Dimethylaniline in 46ml of HCl in 500ml beaker. Add ice pieces and bring the temperature below 5\(^\circ\)C. Dissolve 15gm NaNO\textsubscript{2} in 10 to 15ml water. Add NaNO\textsubscript{2} solution slowly with constant stirring in the above solution to about 30 minutes time. The temperature should not rise above 5\(^\circ\)C. Allow to stand for 30 minutes. Filter and wash with dilute HCl and dry.

CALCULATION:

\[
\% \text{ yield} = \frac{\text{Practical yield}}{\text{Theoretical yield}} \times 100
\]

RESULT:

3. Percentage yield: \(-\)%
EXPERIMENT: 13

AIM: Preparation of Fast Red B.

REQUIREMENT: α-Naphthyl amine, sodium nitrite, R-salt, conc. HCl acid etc.

REACTION:

PROCESS: The 14.3gm α-Naphthyl amine is dissolved in 100cc of water and 15cc Conc. HCl acid in 250ml beaker. The mixture is heated for getting clear solution. The clear solution is cooled to room temperature and then to 5°C. The NaNO₂ solution (7.2gm sodium nitrite dissolved in 20cc of water) is now added in above solution keeping temperature 5°C. Now prepare a solution of 35gm R-salt and 8.5gm of caustic soda in 100ml. water in 500ml. beaker. Add diazo solution in to the above solution slowly keeping temperature 5°C. During addition, the whole is well stirred mechanically. After stirring for one hour the colour is heated to 80°C and salt added until the coloring matter is nearly all precipitated.

PROPERTIES: A brown powder dissolved in water with magenata red colour. Dyed wool red from an acid bath.

CALCULATION:

% yield = (Practical yield/ Theoretical yield) 100

RESULT:

1. Theoretical yield:  -------gm.
3. Percentage yield:  -------%
EXPERIMENT: 14

AIM: Preparation of p-Nitro acetanilide.

REQUIREMENTS: 10 gms Acetanilide, Conc. Sulphuric acid (167cc), 6.25gm Nitric acid (44cc), 5gm Sulphuric acid (28cc)

REACTION:

\[
\begin{align*}
\text{NHCOCH}_3 & \xrightarrow{\text{Conc. H}_2\text{SO}_4, \text{Conc. HNO}_3, 0-5^\circ\text{C}} \rightarrow \\
\text{NHCOCH}_3 & \quad \text{NO}_2
\end{align*}
\]

PROCESS: The acetanilide is dissolved in 30 gm of sulphuric acid contained in a round bottom flask, the temperature not being allowed to rise above 40°C. The solution is cooled to 5-10°C by immersion in ice water and cooled mixture of 6.25 gm of nitric acid with 5gm of Sulphuric acid is added very slowly. After each addition the flask well shaken and cooled in ice waters the temperature being kept below, 15°C. After standing a short time the nitration mixture is poured into about 10 liters of water containing several lumps of ice, when the p-nitro acetanilide separates out. It is filtered off, washed, free from acid, and dried on a porous plate. Yield 11-12 gm.

USES: p-Nitro acetanilide is used for the preparation of p-nitro aniline and p-amino acetanilide.

CALCULATION:

\[\% \text{ yield} = \left( \frac{\text{Practical yield}}{\text{Theoretical yield}} \right) \times 100\]

RESULT:

1. Theoretical yield: \text{--------}gm.
2. Practical yield: \text{--------}gm.
3. Percentage yield: \text{--------}%
EXPERIMENT: 01

AIM: Dyeing of Cotton / Wool using basic dye. (Crystal Violet)

REQUIREMENTS: 1% Tannic acid, 1% Tartarematic solution, 1% dye solution.

PROCESS:

(a) Mordanting: Make up the bath by addition of 3ml of Tannic acid solution and 47ml of water. Weight 1gm of the hank in water and place in the bath. Heat the bath at 50-60°C for 15 minutes. Squeeze without rinsing. Treat the pattern in fresh bath with 1.5ml Tartarematic (1% solution) in 48.5ml water at 40-45°C for 15 minutes. Finally rinse well and dry.

(b) Dyeing: Prepare the dye bath by addition of 10 ml dye solution (0.1%) and 40ml of water. Dye in cold for 30 minutes with good stirring, then raise the temperature slowly to 40-50°C. Maintain the same for 15 minutes. Finally rinse and squeeze and dry.
EXPERIMENT: 02

AIM: Dyeing of Cotton / Wool using basic dye. (Methylene Blue)

REQUIREMENTS: 1% Tannic acid, 3% Ferrous Sulphate solution, 1% dye solution.

PROCESS:
(a) Mordanting: Place the given hank in 100ml FeSO₄ solution (3%) for half an hour, then immediately transfer the hank into another bath containing 6 ml tannic acid solution and 94 ml water. Keep it for 15 min, squeeze without rinsing.
(b) Dyeing: Prepare the dye bath by addition of 10 ml dye solution (0.1%) and 40ml of water. Dye in cold for 30 minutes with good stirring, then raise the temperature slowly to 40-50°C. Maintain the same for 15 minutes. Finally rinse and squeeze and dry.
EXPERIMENT: 3

AIM:  Dyeing of cotton /wool using direct dye. (Congo Red)

REQUIREMENT: 0.1% dye solution, 10% w/v sodium chloride solution.

PROCESS:  Make up the bath by addition of 0.1 % dye solution (10 ml and 40 ml water) wet. The fabric in water places the wet fabric in the dye bath, stir well. Raise the temperature with stirring to 80-85°C in 5 minutes. Maintain the same temperature for 45 minutes. Remove the pattern. Add 2 ml sodium chloride solution (10 % w/v) to the bath. Continue the dyeing for further addition of the hot water. Then remove the pattern, rinse with cold water, squeeze and dry.
EXPERIMENT: 04

AIM: Dyeing of cotton using Aniline Black dye.

REQUIREMENTS: Potassium dichromate solution (10%), copper sulphate solution (2%), hydrochloric solution (10%), aniline hydrochloride.

PROCESS: Prepare a dye bath in a 250cc beaker by addition of the following. 140ml water, 17.5ml potassium dichromate solution (10%), 3.5ml copper sulphate solution (2%), 1ml hydrochloric acid solution (10%). Then add 5gm Aniline hydrochloride introduce the wetted cotton hank in the dye bath, work in cold for 45 minutes. Then raise the temperature slowly to boiling within ½ hrs. Work in hot dye bath for ½ hours, squeeze, rinse and dry.
EXPERIMENT

AIM: DETERMINATION OF NITROGEN BY THE KJELDLH’S METHOD

PRINCIPLE: The nitrogen in the sample is converted to ammonium sulphate by digestion with concentrated H₂SO₄ acid and a suitable catalyst. The digest is made alkaline and the liberated ammonia is separated by steam distillation. The distillate is absorbed in boric acid or nickel ammonium sulphate solution and titrated with standard acid.

N.B.: The method is applicable to compounds containing amino and amino nitrogen and many heterocyclic compounds containing nitrogen and to in the ring. The method fails for compounds containing N-N, NO and NO₂ groups and it is necessary to reduce such compounds before digestion.

Reagents:
(1) Sulphuric acid A.R., (2) Catalyst mixture: 20gm Potassium sulphate, 1gm cupric sulphate, 1gm selenium metal. (3) 40% sodium hydroxide (80gms NaOH solution) 100 gms (4) 0.1 N HCl exact.

Screen indicator: 0.125gm methyl red, 0.083 gms methylene blue in 100 ml ethanol.

Process:
Digestion: About 100 to 150 mgs of the substance to be analyzed is weighed accurately and transferred to a clean and dried kjeldahl digestion flask. Add 2.0gm of the catalyst mixture. Measure out 5.0 ml of the concentrated H₂SO₄ and pour it carefully into the flask. Place a small funnel over it and support the flask in a stand so that it is slightly inclined from the vertical. Heat the mixture over a burner with low flame so that the solution boils gently for five minutes, then increase the heating so that the solution boils vigorously and continue the heating for further 45 minutes; the liquid should be colourless at the end of this period. Allow the digestion mixture in the kjeldhal flask to cool, add 15 ml 50% (w/v) NaOH and dilute it continuously with 10 ml of distilled water. Carry out a parallel blank determination using same quantities of reagents except the nitrogenous compound, this will serve as a test for the purity of the reagents.

Distillation: Arrange the distillation apparatus for the distillation of ammonia and absorb it in saturated boric acid (25 ml) soln. Titrate this soln. against standard 0.1 N HCl using screen indicator. The end point will be green to violet. Note the vol (y ml) of 0.1 N HCl.
OBSERVATION:

EQUATION/REACTION:

Digestion

1. Nitrogen containing compound + H₂SO₄ → (NH₄)₂SO₄

   Ammonium Sulphate

2. (NH₄)₂SO₄ + NaOH → NH₃(g)

Distillation:

3. NH₃ + H₃BO₃ → NH₄⁺ + H₂BO₃⁻

4. H₂BO₃⁻ + HCl → H₂BO₃ + Cl⁻

CALCULATIONS:

1 ml 0.1 N HCl = 0.28 mg N₂, Y ml 0.1 N HCl = 0.028 x Ymg N₂

\[ \% \text{ N} = \frac{0.28 \times Y \times 100}{\text{wt. of the sub}} \]

Or

\[ \% \text{ N} = \frac{100 \times Y \times 14}{\text{Wt. (mg) of sub}} \]
EXPERIMENT: 01

AIM: TO DETERMINE THE NEUTRALIZATION CAPACITY OF GIVEN ANTACID.

REQUIREMENTS: 0.1 N HCl, 0.1 N NaOH, Indicator.

PROCESS: Weigh an antacid tablet accurately to + 0.0002 gm. Transfer it to 250 ml conical flask and add to it exactly 75 ml 0.1 N HCl. Heat the solution to boiling for 5 minutes. Allow it to cool and titrate the solution against 0.1 N NaOH using phenolphthalein as indicator. Perform the blank in the same manner without using sample. Perform the standardization of 0.1 N HCl and 0.1 N NaOH.

CALCULATION:
1. Blank reading = A ml of 0.1 N NaOH
2. Sample reading = B ml of 0.1 N NaOH
3. NaOH consumed = Blank - Sample reading
   = A - B = C ml. NaOH
EXPERIMENT: 02

AIM: TO PERFORM THE ASSAY OF ZnO IP 85.

REQUIREMENT: Sample, NaOH, H\textsubscript{2}SO\textsubscript{4}, KHP, NH\textsubscript{4}CL etc.

PROCESS: Weigh accurately about 1.5 gm & dissolve it in 2.5 gm NH\textsubscript{4}CL and 50 ml of 1 N H\textsubscript{2}SO\textsubscript{4}. Heat the flash gently for few minutes and then titrate against the 1N NaOH using Methyl orange. Perform the Blank experiments also.

EQUATION:
\[
\text{ZnO + H}_2\text{SO}_4 \rightarrow \text{ZnSO}_4 + \text{H}_2\text{O} \\
\text{H}_2\text{SO}_4 + 2\text{NaOH} \rightarrow \text{Na}_2\text{SO}_4 + 2\text{H}_2\text{O}
\]

CALCULATION:
1 ml. 1 N H\textsubscript{2}SO\textsubscript{4} = 0.04069 gm of Zn

RESULT:
% purity of ZnO is _________ % w/w
EXPERIMENT: 03

Aim: To determine the % Aspirin in the given sample.

Requirements: Std. 0.5 N NaOH Soln., Std. 0.5 N HCl Soln., Phenolphthaleine

Process: Accurately weight out around 0.5 gm of given sample and dissolve in a 10 ml of absolute alcohol. Now add 50 ml of std. soln of NaOH by means of burette and then heat the flask on water bath at boiling temperature for 25 min. cool the flask at room temperature. Titrate it aganst with std. soln of HCl using phenolphthaleine as an indicator. Also perform the blank experiment without sample.

Result:
% Aspirin in a given sample = ____________
EXPERIMENT: 04

**Aim:** Determination of the % w/w of lactic acid and lactide together calculated as CH₃CHOHCOOH

**Requirement**
1.0 N NaOH, Lactic acid, 1.0N HCl, phenolphthalein indicator

**Process**

Weigh the sample (3 to 4gms) in a weighing bottle. Transfer the sample into a conical flask, using about 50ml water. Lactic acid is viscous and so the weighing bottle must be washed out carefully. Add 50ml of 1N NaOH. Keep the flask in a boiling water bath for 5 min. then cool the flask under tap water. Back titrate the excess of alkali with 1N HCl using phenolphthalein indicator. Carry out the blank titration with 50ml of 1N NaOH.

**Calculation**

1ml 1N NaOH = 0.090089 gm of lactic acid

**Results**

% of lactic acid = _________