LAB MANUAL
(2016–17)
15 CHEL 17/27 Engineering Chemistry Lab
I/II Semester

Name: _______________________________________________________

U S N: ___________________________ Branch: ________________

Batch: ________________________ Section: ________________
General Chemistry Lab Safety

Conduct:

1. Eating, drinking, and smoking are strictly prohibited in the laboratory.
2. No unauthorized experiments are to be performed.
3. Never taste anything. Never directly smell the source of any vapor or gas; instead by means of your cupped hand, waft a small sample to your nose. Do not inhale these vapors but take in only enough to detect an odor if one exists.
4. Coats, backpacks, etc., should not be left on the lab benches and stools.
5. Always wash your hands before leaving lab.
6. Learn where the safety and first-aid equipment is located. This includes fire extinguishers, fire blankets, and eye-wash stations.
7. Notify the instructor immediately in case of an accident.

Proper Handling of Chemicals and Equipment:

8. Consider all chemicals to be hazardous unless you are instructed otherwise. Material Safety Data Sheets (MSDS) are available in lab for all chemicals in use. These will inform you of any hazards and precautions of which you should be aware.
9. Know what chemicals you are using. Carefully read the label twice before taking anything from a bottle. Chemicals in the lab are marked with NFPA hazardous materials diamond labels. Learn how to interpret these labels.
10. Excess reagents are never to be returned to stock bottles. If you take too much, dispose of the excess.
11. Many common reagents, for example, alcohols and acetone, are highly flammable. Do not use them anywhere near open flames.
12. Always pour acids into water. If you pour water into acid, the heat of reaction will cause the water to explode into steam, sometimes violently, and the acid will splatter.
13. If chemicals come into contact with your skin or eyes, flush immediately with copious amounts of water and consult with your instructor.
14. Never point a test tube or any vessel that you are heating at yourself or your neighbor it may erupt like a geyser.
15. Dispose of chemicals properly.
16. Clean up all broken glassware immediately and dispose of the broken glass properly.
17. Never leave burners/hot plates unattended. Turn them off whenever you leave your workstation. Be sure that the gas is shut off at the bench rack when you leave the lab.
18. Beware of hot glass it looks exactly like cold glass.
SYLLABUS

Engineering chemistry Laboratory
(Common to CS/IS/EC/EE/ME/CV)

Sub Code: 15CHEL17/15CHEL27
IA Marks: 20
Hrs/week: 03
Exam Hours: 03
Total Hours: 50
Exam Marks: 80
Credits: 02

PART – A

1. Potentiometric estimation of FAS using standard K₂Cr₂O₇ solution
2. Colorimetric estimation of Copper
3. Conductometric estimation of an Acid mixture using standard NaOH solution
4. Determination of pKa of a weak acid using pH Meter
5. Determination of Viscosity co-efficient of a given liquid using Ostwald’s Viscometer.
6. Flame Photometric estimation of Sodium and Potassium in the given sample of Water

PART – B

1. Determination of Total Hardness of a sample of Water using Disodium salt of EDTA.
2. Determination of CaO in the given sample of Cement by Rapid EDTA method.
3. Determination of Percentage of Copper in Brass using standard Sodium thiosulphate solution.
5. Determination of COD of the given Industrial Waste water sample.
6. Determination of Total Alkalinity of a given Water Sample using
standard Hydrochloric acid.

**Channabasaveshwara Institute of Technology**
(An ISO 9001:2008 Certified Institution)
**NH 206 (B.H. Road), Gubbi, Tumkur – 572 216. Karnataka.**

**DEPARTMENT OF CHEMISTRY**

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<td>11.</td>
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<tr>
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References
Viva Questions
Questions
Model Procedure
DETERMINATION OF pK\textsubscript{a} OF A WEAK ACID USING pH METER

Aim: To determine pK\textsubscript{a} value of weak acid using digital pH meter

**Principle:** During the titration of an acid with a base, the pH of the solution rises gradually at first, then more rapidly, until at the equivalence point, there is a very sharp increase in pH for every small quantity of base added, once pass the equivalence point, the pH increases only slightly on addition of excess base. The titration curve obtained by plotting changes in pH at different amounts of the base added is given in figure.

According to Henderson-Hasselbalch equation. \( \text{pH} = \text{pK}_a + \log \frac{[\text{salt}]}{[\text{acid}]} \). Where \( K_a \) is dissociation constant of the acid and \( \text{pK}_a = -\log K_a \).

At half equivalence point, \([\text{salt}] = [\text{acid}]\). and hence \( \text{pH} = \text{pK}_a \). Therefore pH at half equivalence point gives the pK\textsubscript{a} of weak acid.

**Procedure:** Pipette out 25ml of the given weak acid into a beaker. Immerse the glass and calomel electrode assembly into it. Connect the electrodes to pH meter and measure the pH of acid. Add NaOH from the burette in increments of 0.5ml and measure the pH after each addition. Plot the graph of \( \Delta \text{pH} / \Delta V \) vs volume of NaOH and determine the equivalence point(X).Plot the another graph of pH vs volume of NaOH and determine the half equivalence point(x/2). Determine the pK\textsubscript{a} value at Half equivalence point.

**Result:** The pK\textsubscript{a} value of a given weak acid is -----------
Observation and Calculation:

<table>
<thead>
<tr>
<th>Volume of NaOH (V)</th>
<th>∆V</th>
<th>pH</th>
<th>∆pH</th>
<th>∆pH/∆V</th>
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Equivalence point, $X = \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldordering point, $X = \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ld..
Experiment No. 2

Date: __ / __ / ______

POTENTIOMETRIC TITRATION-ESTIMATION OF FAS USING STANDARD POTASSIUM DICHROMATE SOLUTION

Aim: Estimation of FAS Potentiometrically using Std. Potassium dichromate solution

Principle: The procedure of using a measurement of emf to determine the concentration of ionic species in solution is called as potentiometry. The principle involved in potentiometric titration is the measurement of emf between two electrodes, an indicator electrode,( the potential of which is function of the concentration of the ion to be determined) and a reference electrode of constant potential . In these titration, the measurement of emf are made while the titration is in progress. The equivalence point of the reaction is revealed by a sudden change in potential in the plot of emf readings against the volume of titratant.

The determining factor in oxidation and reduction reactions is the ratio of the concentration of oxidized and reduced forms of certain species

\[ E_{cell} = E^0 + 0.0591/n \log [Fe^{3+}] / [Fe^{2+}] \]

Where \( E^0 \) = is the standard potential of the system.

The potential of the immersed electrode is controlled by the ratio of these concentrations. During the oxidation of a reducing agent or the reduction of an oxidizing agent, the ratio changes, hence the emf changes more rapidly in the vicinity of the end point.

Procedure:

Pipette out 25ml of the given FAS solution into a beaker. Add one test tube of dilute sulphuric acid. Immerse the Platinum and calomel electrode assembly into it. Connect the electrodes to potentiometer and measure the potential. Add \( \text{K}_2\text{Cr}_2\text{O}_7 \) from the burette in increments of 0.5ml and measure the potential after each addition. Plot a graph of \( \Delta E / \Delta V \) against volume of \( \text{K}_2\text{Cr}_2\text{O}_7 \) and determine the equivalence point. From the normality of \( \text{K}_2\text{Cr}_2\text{O}_7 \), calculate the normality and weight of FAS present in the given solution.

Result: Weight of FAS present in a dm³ of the given solution = ------------------ g
Observation and Calculation:

<table>
<thead>
<tr>
<th>Volume of $\text{K}_2\text{Cr}_2\text{O}_7$ (V)</th>
<th>Emf (E)mv</th>
<th>$\Delta V$</th>
<th>$\Delta E$</th>
<th>$\Delta E / \Delta V$</th>
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</table>

$$N_{\text{K}_2\text{Cr}_2\text{O}_7} x V_{\text{K}_2\text{Cr}_2\text{O}_7}$$

$$N_{\text{FAS}} = \frac{N_{\text{K}_2\text{Cr}_2\text{O}_7} x V_{\text{K}_2\text{Cr}_2\text{O}_7}}{\text{Volume of FAS}}$$

$$0.5 x \frac{N_{\text{FAS}}}{25} = \frac{N_{\text{FAS}}}{25} = 25$$

Weight of FAS present in a dm$^3$ of the given solution = $N_{\text{FAS}}$ x Eq.weight of FAS (392.14)

$$= \frac{N_{\text{FAS}}}{25} = 25$$

Weight of FAS present in a 25 Cm$^3$ of the given solution = $\frac{N_{\text{FAS}}}{1000} x 25$
COLORIMETRIC DETERMINATION OF COPPER

Aim: Estimation of Copper colorimetrically

Principle:

When a monochromatic light of intensity $I_o$ is incident on a transparent medium, a part $I_a$ of it is absorbed, a part $I_r$ is reflected & the remaining part $I_t$ is transmitted.

$$I_o = I_a + I_r + I_t$$

For a glass-air interface $I_r$ is negligible.

Therefore $I_o = I_a + I_t$

$I_t / I_o = T$ called the transmittance $\log \frac{I_o}{I_t}$ is called the absorbance or optical density.

The relation between absorbance $A$, concentration $C$ & path length $I$ is given by Beer-Lambert’s law.

$$A = \log \frac{I_o}{I_t} = \varepsilon Ct$$

Where ‘$\varepsilon$’ is the molar extinction coefficient, ‘$t$’ is the path length. ‘$C$’ is a constant for a given substance at a given wave length. If $t$ is the path length is kept constant, then $A \alpha C$. Hence a plot a graph of absorbance against concentration gives a straight line.

In a colorimeter, a narrow beam of light of proper wavelength passes through the test solution, towards a sensitive photocell (detector). Generally, a colorimeter is provided with arrangement of filter to select the light of required wavelength. The current generated in the photocell is proportional to the amount of light transmitted by the solution. This inturn depends upon the concentration of test solution. Thus, the current from the photocell will be more when the light transmitted by the solution is more. This generally happens when the test solution is more dilute.

A series of std. solutions of copper is treated with ammonia to get blue Cuprammonium complex $[\text{Cu(NH}_3\text{)}_4]^2+$ and is diluted to a definite volume. The absorbance of each these solutions is measured at 620 nm since the complex shows maximum absorbance at this wavelength. The absorbance values are plotted against volume of copper sulphate to get a calibration curve.

A known volume of the test solution is treated with ammonia & diluted up to the mark. The absorbance of this solution at 620 nm is measured & its concentration is determined from the calibration curve.
Procedure:
Transfer the given copper sulphate solution (stock solution) to a burette and draw out 2, 4, 6, 8 & 10 ml of given copper sulphate solution into 5 separate 50 ml volumetric flasks. Now collect the test solution from the teacher/examiner, add 5 ml of ammonia solution to each of them and shake well and then dilute up to the mark with distilled water and mix well. Prepare a blank solution by diluting only 5 ml of ammonia solution in a 50 ml measuring flask up to the mark with distilled water and mix well. After 10 min, measure the absorbance of the solution against blank adjusting to 0.0 absorbance at 620 nm using a photoelectric colorimeter. Tabulate the readings as shown. Draw a calibration curve by plotting absorbance against volume of copper sulphate solution. Using the calibration curve, find out the volume of copper sulphate solution given i.e., the volume of test solution and calculate the amount of copper in the given solution.

(Note: use distilled water only)

Result: The given test solution contains -------- mg of Cu

Observation and calculation:

<table>
<thead>
<tr>
<th>Volume of copper Sulphate (ml)</th>
<th>Concentration of copper = 2.037 mg x volume of copper sulphate solution</th>
<th>Absorbance (Optical density)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Blank Solution</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>2.0</td>
<td>2.037 mg x 2 =</td>
<td></td>
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<tr>
<td>4.0</td>
<td>2.037 mg x 4 =</td>
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<tr>
<td>6.0</td>
<td>2.037 mg x 6 =</td>
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<tr>
<td>8.0</td>
<td>2.037 mg x 8 =</td>
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<tr>
<td>10.0</td>
<td>2.037 mg x 10 =</td>
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</tr>
<tr>
<td>Test Solution</td>
<td>2.037 mg x a =</td>
<td></td>
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</tbody>
</table>

From graph, volume of the given test solution, a = ------------------- ml
Copper present in the given test solution = 2.037 mg x volume of test solution (‘a’ ml)

= 2.037 mg x --------

= ------------------- mg
Experiment No. 4

Date: __ /__ / _____

CONDUCTOMETRIC ESTIMATION OF ACID MIXTRE USING STD. NaOH SOLUTION.

Aim: Estimation of acid mixture conductometrically using std. NaOH solution

Principle: Measurement of conductance can be employed to determine the end point in acid –base titrations. In conductometric titrations there is a sudden increase in conductance of the solution at the equivalence point. The principle underlying conductometric titrations is the substitution of ions of one mobility by ions of other mobility. Therefore, the conductance of a solution depends on the number & mobility of ions. The equivalence point is determined graphically by plotting conductance against titer values.

Conductometric titration may be applied for the determination of acid mixture. In this titration, on adding a strong base to a mixture of a strong acid and a weak acid, the conductance falls till a strong acid is neutralized completely due to removal of $H^+$ ions. The weak acid remains undissociated in the presence of strong acid. Once the strong acid is completely neutralized, the weak acid begins to dissociate and gets neutralized. This results in increase of conductance of solution. When the neutralization of second acid is complete, their will be steep increase in conductance due to the ions furnished by strong base.

Procedure: Pipette out 25ml of acid mixture in a beaker. Immerse the conductivity cell in the given solution and connect it to conductivity meter. Stir the solution and measure the initial conductance of the solution. Add 1N NaOH from the burette in the increments of 0.5 ml after each addition stir the solution and note down the conductance. continue the titration till decreasing trend changes to increasing trend.

Plot the graph of conductance on Y-axis and volume of NaOH on X-axis to get two straight lines. The point of intersection of two straight lines gives the equivalence point. The equivalence point corresponds to volume of NaOH required neutralising HCl. By knowing the normality & volume of NaOH calculate the normality & amount of HCl & CH$_3$COOH.

Report:: The amount of HCl present in the given solution ______g

The amount of CH$_3$COOH present in the given solution______g
**Calculation:**

Normality of NaOH = 1 N

Volume of NaOH required to neutralize HCl = \( V_1 \) cm³

Normality of HCl = \( \frac{\text{Normality of NaOH} \times \text{Volume of NaOH}}{\text{Volume of HCl}} \)

\[ \frac{1 \times V_1}{50} = \text{N} \]

The weight of HCl dm⁻³ = Normality x Eq. Wt. of HCl

\[ \frac{\text{Normality} \times 36.5}{\text{Volume of NaOH (ml)}} \]

\[ \frac{\text{Normality} \times 60}{\text{Volume of NaOH (ml)}} \]

<table>
<thead>
<tr>
<th>Volume of NaOH (ml)</th>
<th>Conductance (S/m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td></td>
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<tr>
<td>0.5</td>
<td></td>
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<tr>
<td>1.0</td>
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<td>1.5</td>
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<tr>
<td>19.0</td>
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<tr>
<td>20.0</td>
<td></td>
</tr>
</tbody>
</table>


Experiment No. 5

DETERMINATION OF VISCOSITY COEFFICIENT OF A GIVEN LIQUID USING
OSTWALD’S VISCOMETER

Aim: To determine the viscosity coefficient of a given liquid using ostwalds viscometer

Principle:
Viscosity arises due to internal friction between moving layer of molecules. A liquid flowing through cylindrical tube of uniform diameter is expected to move in the form of molecular layers. A layer close to surface is almost stationary while that at the axis of the tube moves faster than any other intermediate layer. A slow moving layer exerts a drag or friction on its nearest moving layer backwards. This property of a liquid by which it retards or opposes motion between layers is called viscosity. The coefficient of viscosity is defined as the tangential force per unit area required to maintain a unit velocity gradient between any to successive layers of a liquid situated unit distance apart. The coefficient of viscosity of a liquid is given by Poiseuille’s formula.

\[
V = \frac{\Pi \rho r^4 t}{8 \eta l}
\]

Where \( \rho = hgd \)

\( V \) = volume of the liquid, \( r \) = radius of the tube, \( l \) = length of the tube, \( \rho \) = pressure difference between two ends of the tube, \( \eta \) = the coefficient of viscosity of the liquid. If equal volumes of two different liquids are allowed to flow through the same tube under identical conditions then,

\[
\eta_L = \frac{\eta_w x t_L x d_L}{t_w x d_w}
\]

The time \( t_L \) taken by the given liquid to travel through a certain distance in the tube is determined. The time, \( t_w \) taken by a standard liquid to travel through the same distance is measured. Knowing the densities \( d_L \) and \( d_w \) of the test liquid and the standard and also the coefficient of viscosity of the standard (\( \eta_w \)), coefficient of viscosity of the test liquid (\( \eta_L \)) can be calculated.

Procedure:

Pipette out 20 ml of given liquid into a wide limb of viscometer (fixed vertically to the stand) and suck the liquid through the other limb. Determine the time flow between 2 fixed points. One above & one below the bulb in the narrow limb of viscometer. Repeat and calculate the average time flow. Pour out the liquid, wash the viscometer with acetone and dry it. Now pipette out 20ml of water into limb and determine the average time flow for water as before. Determine the coefficient of viscosity of the given liquid using the formula.

Result: The coefficient of viscosity of the given liquid = ............... Millipoise.
Observation and calculation:

Laboratory Temperature = .................

$\eta_w$ = Viscosity coefficient of water = ...... millipoise

$\eta_L$ = Viscosity coefficient of liquid = ?

$d_w$ = density of water = .............. g/cc

$d_L$ = density of liquid = .............. g/cc

\[
\eta_L = \frac{\eta_w \times t_L \times d_L}{t_w \times d_w}
\]

= .............. millipoise.

The coefficient of viscosity of the given liquid = .............. millipoise.
FLAME PHOTOMETRIC ESTIMATION OF SODIUM IN THE GIVEN SAMPLE OF WATER

Aim: To estimate the amount of sodium and potassium in the given sample of water using flame photometer

Principle:
Flame photometry is an atomic emission technique used for detection of metals. If a solution containing metallic salts is aspirated into a flame, a vapor, which contains metallic atoms, will be formed. The electrons from the metallic atoms are then excited from ground state ($E_1$) to higher energy state ($E_n$) where $n = 2, 3, 4$ ------7, by making use of thermal energy of flame. From higher energy states these electrons will return to the ground state by emitting radiations ($E_n - E_1 = h\nu$, where $n = 2, 3, 4$---7), which are the characteristic of each element.

\[
\text{NaCl (s)} \rightarrow \text{NaCl (g)} \xrightarrow{\text{Dissociation}} \text{Na (g)} + \text{Cl (g)} \\
\text{KCl (s)} \rightarrow \text{KCl (g)} \xrightarrow{\text{Dissociation}} \text{K (g)} + \text{Cl (g)}
\]

Flame photometer correlates the emitted radiations with the concentration of these elements. It is simple and rapid method for the elements that can be easily excited (Sodium, Potassium and other alkali metals)

A flame photometer is composed of the pressure regulator and flow meter for fuel gases an atomizer, burner, optical system, photosensitive detector and output recorder. A filter of the element whose concentration is to be determined is inserted between the flame and the detector. Propane gas is used as fuel and air or oxygen is used as oxidant. Combination of these two will give a temperature of 1900° C. The whole analysis depends on the flow rate of the fuel, oxidant, the rate of introduction of the sample and droplet size.
The sample containing the analyte is aspirated into the flame through atomizer. Radiation from resulting flame is collected by the lens and allowed to pass through an optical filter, which permits only the radiation characteristic of the element under investigation into the photocell. The output from the photocell represents the concentration and nature of the element.

**Procedure:**

Prepare the standard solution of sodium and potassium of concentration 2, 4, 6, 8 & 10 ppm by transferring 2, 4, 6, 8, 10 ml of stock solution (50 ppm) into different 50ml volumetric flask using a burette. Dilute all the solutions up to the mark using distilled water, dilute the test solution of unknown concentration, and shake well for uniform concentration. Place the distilled water in the suction capillary of the instrument and set the instrument to read zero. Place each of the standard solutions in the suction capillary and set the instrument to read the flame emission intensity 2, 4, 6, 8 and 10 respectively using sodium filter (598 nm) (Rinse with distilled water after each reading) Dilute the given test solution up to the mark and mix well. Place the test solution in the suction capillary and record the reading. Draw a calibration curve by plotting the emission intensity on y-axis and volume of NaCl solution on X-axis. From the calibration curve, find out the volume of the given test solution and calculate the amount of sodium in the water sample.

**Result:** The given test solution contains -------- ppm of Na

**Observation and calculation:**

<table>
<thead>
<tr>
<th>Volume of NaCl (ml)</th>
<th>Concentration of NaCl in ppm</th>
<th>Emission Intensity (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0</td>
<td>-----</td>
<td></td>
</tr>
<tr>
<td>2.0</td>
<td>0.02</td>
<td></td>
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<tr>
<td>4.0</td>
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<tr>
<td>6.0</td>
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</tr>
<tr>
<td>8.0</td>
<td>0.08</td>
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<td>10.0</td>
<td>1.0</td>
<td></td>
</tr>
<tr>
<td>Test solution</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

58.5g of NaCl contains 23.0g of Na

\[
X \text{ ppm of NaCl contains } (1000 \text{ ml}) = \frac{X \times 23}{25} = \text{-----ppm}
\]
Experiment No. 7  

DETERMINATION OF PERCENTAGE OF COPPER IN BRASS USING STANDARD SODIUM THIOSULPHATE SOLUTION.

Aim: To determine the percentage of Copper in brass using Std. sodium thiosulphate solution

Principle:
The chief constituents of brass alloy are copper and zinc. It also contains small quantities of tin, lead & iron. The percentage composition of a typical brass alloy is Cu: 50 – 90 %, Zn: 20 -40 %, Sn : 0 – 6 % , Pb – 2 % , Fe : 0 – 1 %.

A solution of brass is made by dissolving the brass sample in minimum amount of nitric acid; excess of nitric acid is destroyed by boiling with urea. The solution is neutralized & made faintly acidic. Potassium iodide solution is added, the iodine liberated (cupric ions oxidizes iodide to iodine), is titrated against sodium thiosulphate using starch as indicator.

\[
2\text{Cu}^{2+} + 4\text{KI} \rightarrow \text{Cu}_2\text{I}_2 + 4\text{K}^+ + \text{I}_2
\]

\[
2\text{S}_2\text{O}_3^{2-} + \text{I}_2 \rightarrow \text{S}_4\text{O}_6^{2-} + 2\text{NaI}
\]

Procedure:

PART A: Preparation of brass solution

Weigh out the given brass sample into a clean beaker. Add ¼ test tube con. HNO₃ till brass dissolves completely. Add 1 test tube of distilled water and 1 gm of urea, boil till all brown fumes are expelled. Cool to room temperature. transfer the solution in to a 250ml volumetric flask and dilute up to the mark with distilled water and mix well.

PART B: Estimation of copper in brass solution

Pipette out 25ml of brass solution into a clean conical flask; add ammonium hydroxide drop by drop until a pale blue precipitate is obtained. Dissolve the precipitate by adding 1/4th test tube of dilute acetic acid. Add 1 test tube of 10% KI solution .Titrate the liberated I₂ against standard Na₂S₂O₃ solution until the solution becomes pale yellow. Add 2 Cm³ of freshly prepared starch as an indicator, continue the titration till the blue color disappears leaving behind white precipitate. Repeat the titration till the concordant values are obtained.

RESULT: Percentage of Copper in the given brass sample = ------------------
Observation:

PART A: Preparation of brass solution

Burette : Std. Sodium trio sulphate solution
Conical Flask : 25 ml of Brass solution + NH₄OH drop wise till bluish
White ppt. + ¼ t.t. dil CH₃COOH + 1 t.t. 10 % KI.
Indicator : Starch - added towards the end point.(Pale yellow)
Till the blue colour appears

Colour Change : Blue to white ppt.

PART B: Estimation of copper in brass solution

<table>
<thead>
<tr>
<th>Burette reading</th>
<th>I</th>
<th>II</th>
<th>III</th>
</tr>
</thead>
<tbody>
<tr>
<td>Final burette reading</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Initial burette reading</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Volume of Na₂S₂O₃ run down (ml)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Calculation:
Normality of Sodium thiosulphate $N₁ = 0.05 \text{ N}$

Weight of brass taken ($w$) = --------------- g

Volume of Sodium thiosulphate consumed $V₁ = -------------- \text{ Cm}³$

$1000 \text{ Cm}³$ of $1 \text{ N}$ Sodium thiosulphate = $63.54 \text{ g of Cu}$

Normality of Cu²⁺ solution, $N₂ = \frac{V₁ \times N₁}{25}$

----------

weight of copper in a liter of the solution, $W₃$=Normality of Cu²⁺ $\times$ Eq. wt of Cu

$= N₂ \times 63.54$

$= \text{ ............g}$

$W₃$

Weight of copper in 250 ml of the solution, $W₄ = \frac{W₃}{4}$

$= \text{ ............g}$

$W₄$

Percentage of copper in brass

$= \frac{W₄ \times 100}{\text{ wt .of brass}}$

$= \frac{W₄ \times 100}{W}$

$= \text{ ............}$
Experiment No. 8

PREPARATION OF STANDARD POTASSIUM DICHROMATE SOLUTION AND DETERMINATION OF IRON IN THE GIVEN SAMPLE OF HAEMATITE ORE SOLUTION. (External indicator method)

Aim: To determine the percentage of iron in the given sample of heamatite ore solution by external indicator method.

Haematite is an important ore of iron containing mainly Fe$_2$O$_3$ and small amount of silica (SiO$_2$).

Principle:
Haematite is an important ore of iron containing mainly Fe$_2$O$_3$ and small amount of silica (SiO$_2$). Iron is present in the solution as Fe$^{3+}$ ions. It is reduced to Fe$^{2+}$ ions using stannous chloride in hot condition in presence of concentrated HCl. The excess of stannous chloride added is oxidized by treating the solution with mercuric chloride. The ferrous ions in the resulting solution are treated against standard potassium dichromate using potassium ferricyanide as external indicator. Potassium ferricyanide produces an intense blue colour with ferrous ions due to the formation of ferriferrocyanide complex.

\[
\text{Fe}_2\text{O}_3 + 6\text{HCl} \rightarrow 2\text{FeCl}_3 + 3\text{H}_2\text{O}
\]
\[
2\text{FeCl}_3 + \text{SnCl}_2 \rightarrow 2\text{FeCl}_2 + \text{SnCl}_4
\]
\[
\text{SnCl}_2 + 2\text{HgCl}_2 \rightarrow \text{Hg}_2\text{Cl}_2 + \text{SnCl}_4
\]
\[
\text{Fe}^{2+} \rightarrow \text{Fe}^{3+} + \text{e} \quad \text{x 6} \quad \text{------- (1)}
\]
\[
\text{Cr}_2\text{O}_7^{2-} + 14\text{H}^+ + 6\text{e} \rightarrow 2\text{Cr}^{3+} + 7\text{H}_2\text{O} \quad \text{--- (2)}
\]

Adding (1) & (2)
\[
6\text{Fe}^{2+} + \text{Cr}_2\text{O}_7^{2-} + 14\text{H}^+ \rightarrow 6\text{Fe}^{3+} + 2\text{Cr}^{3+} + 7\text{H}_2\text{O}
\]

Procedure:
Part A: Preparation of standard K$_2$Cr$_2$O$_7$ solution
Weigh out 0.5g of K$_2$Cr$_2$O$_7$ crystals accurately and transfer into a 250ml volumetric flask. Dissolve in distilled water and dilute up to the mark, mix well.

Part B: Estimation of Iron
Pipette out 25cm$^3$ of the haematite ore solution into a clean conical flask. Add a quarter test tube of concentrated HCl and heat the solution to boiling. Add stannous chloride to the hot solution drop wise till the yellow solution turns colorless .Add 2 more drops to ensure complete reduction. Cool and add a quarter test tube of mercuric chloride (silky white ppt is formed). Add one test tube of water. Place a number of drops of freshly prepared potassium ferricyanide indicator on the wax paper. Add a small quantity of K$_2$Cr$_2$O$_7$ from burette to the conical flask containing haematite solution and mix well. Take a drop of the solution from the conical flask and bring it in contact with a drop of the indicator on wax paper. The color of the indicator turns blue. Repeat this after adding 1cm$^3$ more of the K$_2$Cr$_2$O$_7$ solution .The indicator turns blue as long as the titration is incomplete. Continue the titration by adding increment of 1cm$^3$ of K$_2$Cr$_2$O$_7$ at a time and testing as above till a drop of mixture fails to produce any color with the indicator drop. (Note: Clean the glass rod after every test). Repeat the titration by taking another 25 Cm$^3$ of the haematite solution. This time add most of the K$_2$Cr$_2$O$_7$ solution required at a stretch and then drop wise. Mix the
contents of the flask after every addition and test a drop of the titrated mixture with a drop of the indicator as described above till the color of the indicator drop does not change. Repeat for concordant values.

**RESULT:** Percentage of iron in the given haematite sample = ----------------------

**Observation:**

**Part A: Preparation of standard \( \text{K}_2\text{Cr}_2\text{O}_7 \) solution**

<table>
<thead>
<tr>
<th>Weight of weighing bottle + ( \text{K}_2\text{Cr}_2\text{O}_7 ) crystals (W1)</th>
<th>= -------------- g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Weight of empty weighing bottle (W2)</td>
<td>= -------------- g</td>
</tr>
<tr>
<td>Weight of ( \text{K}_2\text{Cr}_2\text{O}_7 ) crystals transferred (W1 – W2)</td>
<td>= -------------- g</td>
</tr>
</tbody>
</table>

\[
\text{Normality of } \text{K}_2\text{Cr}_2\text{O}_7 = \frac{\text{Weight of } \text{K}_2\text{Cr}_2\text{O}_7 \times 4}{\text{Equivalent weight of } \text{K}_2\text{Cr}_2\text{O}_7 (49)}
\]

**Part B: Estimation of Iron**

**Burette** : Std. \( \text{K}_2\text{Cr}_2\text{O}_7 \) Solution

**Conical Flask** : 25 ml Haematite ore solution + 1/4\(^{th}\) t.t. of con. HCl, boil + \( \text{SnCl}_2 \) drop wise till colorless. Cool & add 1/4\(^{th}\) t.t. \( \text{HgCl}_2 \)

**Indicator** : Potassium ferricyanide as external indicator.

**Color Change**: No change in the color of indicator.

<table>
<thead>
<tr>
<th>Burette reading</th>
<th>I</th>
<th>II</th>
<th>III</th>
</tr>
</thead>
<tbody>
<tr>
<td>Final burette reading</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Initial burette reading</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Volume of ( \text{K}_2\text{Cr}_2\text{O}_7 ) run down in Cm(^3)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Calculation:**

Volume of \( \text{K}_2\text{Cr}_2\text{O}_7 \) consumed = --------------

Weight of Haematite ore in 25 Cm\(^3\) = 0.1 g

1000 Cm\(^3\) of 1N \( \text{K}_2\text{Cr}_2\text{O}_7 \) \( \equiv \) 55.85 g of Fe (1 equivalent of Fe)

\[
\frac{\text{------Cm}^3 \text{ ------N } \text{K}_2\text{Cr}_2\text{O}_7 \equiv \text{55.85 x ------\text{x ------}}}{}
\]

1000 x1

\( \equiv \text{------g of Fe / 25 Cm}^3 \text{ of haematite solution} \)

Percentage of iron in the given haematite sample = \( \text{---------g } \times 100 \)

Weight of ore = ----------------------
Experiment No. 9  

Date: __ / __ / ______

PREPARATION OF STANDARD EDTA SOLUTION AND DETERMINATION OF TOTAL HARDNESS OF A SAMPLE OF WATER

Aim: To determine the total hardness of sample of water using Std. EDTA solution

Principle:

Water with a high concentration of Mg$^{2+}$ and Ca$^{2+}$ is referred to as "hard" water, and the degree of hardness is linked to the concentrations of these two cations [public drinking water: 60-150 mg of CaCO$_3$/L]. Water with a low concentration of these ions is referred to as being "soft". Domestic water is "softened" by exchanging sodium ions for the calcium and magnesium ions.

The hardness of a water sample can be determined by titration of the alkaline earth cations with ethylenediaminetetraacetic acid (EDTA), a complexing agent. The acid itself is practically insoluble in water so it is typical to use the disodium salt of EDTA ("Na$_2$H$_2$Y").

As a complexing agent EDTA has the potential to bind at 6 sites (we say it is hexadentate). It quantitatively forms stable water soluble 1:1, complexes with metal ions (Mg$^{2+}$ and Ca$^{2+}$ : EBT-Mg complex is more stable than that of EBT-Ca complex). Thus EDTA can be used as a titrant to determine the hardness of water. EBT is used as the indicator, the end point being indicated by change in the color from the wine red to blue. Accurate determination of total hardness using EDTA/EBT depends on sharpening the transition from the wine red color of the EBT-complex to the blue color of the uncomplexed EBT.

When added to the solution containing metal ions buffered to a pH of about 10, EBT forms a wine red colored complex with the metal ions (see the below structure).

![EDTA structure](image)

When the solution is titrated with EDTA, EDTA combines with the free metal ions (calcium ions in preference to magnesium ions) present in the solution. At the end point, when all the free metal ions are exhausted added EDTA dissociates the wine red colored metal-EBT complex, consumes the metal ion and releases the blue colored free indicator, hence the color change from red wine to blue.
Procedure:

**Part A: Preparation of standard solution of Disodium salt of EDTA**

Weigh out the given EDTA crystals accurately into a 250ml volumetric flask. Add quarter test tube of ammonia. Dissolve in distilled water and dilute up to the mark, mix well.

**Part B: Determination of total hardness of water sample**

Pipette out 25 Cm$^3$ of the given water sample into a clean conical flask. Add 3ml of Ammonia – ammonium chloride buffer and 1-2 drops of EBT indicator. Titrate against standard EDTA solution till the colour change from wine red to clear blue. Perform the titration slowly near end point. Repeat the titration to get concordant values.

**RESULT:** The total hardness of given water sample = -------------- ppm of CaCO$_3$

**Observation:**

**Part A: Preparation of standard solution of Disodium salt of EDTA**

Weight of weighing bottle + EDTA salt (W1) = ----------------- g

Weight of empty weighing bottle (W2) = ----------------- g

Weight of EDTA transferred (W1 – W2) = ----------------- g

\[
\text{Weight of EDTA taken X 4} \\
\text{Molarity of EDTA} = \frac{\text{Weight of EDTA taken X 4}}{\text{Molecular weight of EDTA (372)}} \\
= \text{------------------- M}
\]
Part B: Determination of Total hardness of water sample

<table>
<thead>
<tr>
<th>Burette reading</th>
<th>I</th>
<th>II</th>
<th>III</th>
</tr>
</thead>
<tbody>
<tr>
<td>Final burette reading</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Initial burette reading</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Volume of EDTA run down (ml)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Calculation:**

Volume of EDTA consumed = ---------------

\[
\text{1000 cm}^3 \text{ of 1 M EDTA} = 100 \text{ g of CaCO}_3 \text{ (Molecular mass of CaCO}_3 = 100) \]

\[
\text{-----} \times \text{-----} \times 100 \]

\[
\text{-----ml of -----M EDTA} = \frac{\text{---------------------}}{1000 \times 1} \text{ g of CaCO}_3
\]

25 cm\(^3\) of the hard water sample contains \------ g of CaCO\(_3\)

\[
\text{10}^6 \text{ cm}^3 \text{ of the hard water sample contains} = \frac{\text{------------}}{25} \text{ ppm of CaCO}_3
\]

Thus total hardness of given water sample = -------------- ppm of CaCO\(_3\)
Experiment No. 10  

PREPARATION OF STANDARD EDTA SOLUTION AND DETERMINATION OF EQUIVALENT OF CALCIUM OXIDE IN THE GIVEN SAMPLE OF CEMENT SOLUTION. (Rapid EDTA Method)

Aim: To determine the percentage of calcium oxide in the given sample of cement solution using Std. EDTA solution

Principle:
Cement contains compounds of Calcium, Aluminium, Magnesium, Iron and insoluble silica. When dissolved in acid, silica remains undissolved. On treating with ammonia, aluminium and iron can be precipitated as their hydroxides and separated. The provided cement solution contains calcium and magnesium ions. The constituents of Portland cement are CaO (60-67%), SiO₂ (17-25%), Al₂O₃ (3-8%), Fe₂O₃ (0.5-6%), MgO(0.1 -4%).SO₃(1-3%), K₂O & Na₂O (0.5-1.5%) , CaSO₄ (3-5%)

To estimate the calcium content in the given solution, a known volume of cement solution is titrated with standard EDTA solution in presence of Mg. Calcium ions present in the cement solution can be titrated against EDTA using Patton & Reader’s indicator in the pH range 12-14. The indicator combines with Calcium ions to form a wine red coloured complex. Ca²⁺ + Indicator Calcium-Indicator complex (Wine red)

Near the end point, when free calcium ions are exhausted in the solution, further addition of EDTA, dissociates Calcium-Indicator complex, consumes the calcium ions and release free indicator which is blue in colour. Therefore colour change is wine red to blue.

Procedure:
Part A: preparation of standard solution of Disodium salt of EDTA

Weigh out the given EDTA crystals accurately into a 250 ml volumetric flask. Add quarter test tube of ammonia. Dissolve in distilled water and dilute up to the mark, mix well.

Part B: Estimation of CaO in the given cement solution

Pipette out 25 Cm³ of the given Cement solution into a clean conical flask. Add 5ml of glycerol, 5ml of diethyl amine and 10ml of 4N NaOH solution. Add 3-4 drops of Patton and Reeder’s indicator. Titrate against standard EDTA solution till the colour change from wine red to clear blue. Repeat the titration to get concordant values.

RESULT: CaO in the given sample of cement solution = -----------------------
Observation:

Part A: preparation of standard solution of Disodium salt of EDTA

Weight of weighing bottle + EDTA salt (W1) = --------------- g

Weight of empty weighing bottle (W2) = --------------- g

Weight of EDTA transferred (W1 – W2) = --------------- g

\[
\text{Weight of EDTA taken } \times 4
\]

\[
\text{Molarity of EDTA} = \frac{\text{Weight of EDTA transferred}}{4 \times \text{Molecular weight of EDTA (372)}}
\]

Part B: Estimation of CaO in the given cement solution

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Calculation:

Volume of EDTA consumed by 25 Cm³ of cement solution = -------------------

Weight of Cement in 25ml =0.1g

1000 Cm³ of 1 M EDTA = 56.08 g of CaO (Molecular mass of CaO =56.08)

\[
\text{------ Cm}^3 \text{ of ------ M EDTA} = \frac{\text{------ g of CaO}}{1000 \times 1} \times 56.08
\]

\[
\text{------ Cm}^3 \text{ of ------ M EDTA} = \frac{\text{------ g of CaO}}{56.08}
\]

25 Cm³ of the Cement solution contains -------- g of CaO

\[
\% \text{ of CaO in the given sample of cement solution} = \frac{\text{------ x 100}}{0.1}
\]
DETERMINATION OF CHEMICAL OXYGEN DEMAND (COD) OF THE INDUSTRIAL WASTE WATER SAMPLE

Aim: To determine the COD of industrial waste water sample

Principle:
The chemical oxygen demand test is extensively employed as a means of measuring the pollutional strength of industrial wastes. Chemical oxygen demand is a measure of the total quantity of oxygen required for complete oxidation of organic compounds of wastes to CO$_2$ and water by a strong oxidizing agent. This parameter is particularly valuable in surveys designed to determine the losses to sewer systems. Results may be obtained within a relatively short time and measures taken to correct errors on the day they occur.

Waste water contains organic impurities which include straight chain aliphatic compounds, aromatic hydrocarbons, straight chain alcohols, acids and other oxidisable materials. Straight chain compounds, acetic acid, etc., are oxidized more effectively when silver sulphate is added as a catalyst. But silver sulphate reacts with chlorides in the waste water to form precipitates which are oxidized only partially by this procedure. This difficulty is overcome by adding mercuric sulphate to the sample.

Procedure:

Preparation of standard FAS solution (Mohr’s salt solution)

Weigh out the given FAS crystals accurately into a 250 ml volumetric flask. Add 2 test tube of H$_2$SO$_4$. Dissolve in distilled water and dilute upto the mark, mix well.

Back titration:

Pipette out 25ml of the waste water sample into a conical flask. Add 10ml of standard potassium dichromate solution followed by 30ml of 6N sulphuric acid (containing silver sulphate) with constant shaking. Add boiling chips to the flask. Attach a reflux condenser and reflux the mixture for half an hour. Cool, wash the condenser with ion exchange water and transfer the washings to the flask. Add 2-3 drops of ferroin indicator and titrate against standard Mohr’s salt solution until the solution turns from blue green to reddish brown. Repeat for agreeing values.

Blank Titration:

Pipette out 10ml of standard potassium dichromate solution. Add 30 ml of 6N Sulphuric acid (containing silver sulphate), reflux for half an hour. Cool and add 2-3 drops of ferroin indicator. Titrate against standard Mohr’s salt solution until the colour turns from blue green to reddish brown.
Observation:
Weight of weighing bottle + FAS crystals \( (W_1) \) = \( \_\_\_\_\_\_\_\_\_ \) g
Weight of empty weighing bottle \( (W_2) \) = \( \_\_\_\_\_\_\_\_\_ \) g
Weight of FAS crystals transferred \( (W_1 - W_2) \) = \( \_\_\_\_\_\_\_\_\_ \) g

Weight of FAS taken \( \times 4 \)
Normality of FAS = \( \frac{\text{Equivalent weight of FAS (392)}}{\text{Burette reading I II III}} \)

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Calculation:
Volume of FAS consumed in Back titration = \( \_\_\_\_\_\_\_\_\_ \) \( \text{Cm}^3 \) \( (Y \text{ Cm}^3) \)
Volume of FAS consumed in Blank titration value = \( \_\_\_\_\_\_\_\_\_ \) \( \text{Cm}^3 \) \( (X \text{ Cm}^3) \)

Volume of FAS consumed for oxidizing organic mater \( (X-Y) \) = \( \_\_\_\_\_\_\_\_\_ \) \( \text{Cm}^3 \)

\(1000 \text{ Cm}^3 \) of \( 1 \text{ N FAS solution} = \) 1 equivalent of oxygen = \( 8 x 10^3 \) mg of oxygen

\( (X-Y) \text{ Cm}^3 \) of \( N_{\text{FAS}} \) solution
\[ = \frac{8 \times 10^3 x (X-Y) x N_{\text{FAS}}}{1000} \]
\[ = \frac{8 \times 10^3 x------}{1000} \]
\[ = \_\_\_\_\_\_\_\_\_ \text{mg of oxygen} \]

25 \( \text{ Cm}^3 \) of waste water sample contains \( \_\_\_\_\_\_\_\_\_ \) mg of oxygen

\(1000 \text{ Cm}^3 \) of waste water sample
\[ = \_\_\_\_\_\_\_\_\_ \times 1000 \]
\[ = \frac{\_\_\_\_\_\_\_\_\_ \text{mg of oxygen}}{25} \]
\[ = \_\_\_\_\_\_\_\_\_ \text{mg of oxygen/dm}^3 \]

COD of waste water sample
\[ = \_\_\_\_\_\_\_\_\_ \text{mg of oxygen/dm}^3 \]

RESULT: The COD of given waste water sample contains \( \_\_\_\_\_\_\_\_\_ \) mg of \( O_2 / \text{dm}^3 \)
DETERMINATION OF TOTAL ALKALINITY OF GIVEN WATER SAMPLE USING
STANDARD HYDROCHLORIC ACID

Aim: To determine the total alkalinity of given water sample using Std. HCl solution

Principle:

The alkalinity of water is due to the presence of hydroxide ion, carbonate ion, and bicarbonate ion present in the given sample of water. These ions can be estimated separately by titrating against standard acid, using phenolphthalein and methyl orange indicator.

The chemical reactions involved are

\[ a) \quad \text{OH}^- + \text{H}^+ \rightarrow \text{H}_2\text{O} \quad \text{(1)} \]
\[ b) \quad \text{CO}_3^{2-} + \text{H}^+ \rightarrow \text{HCO}_3^- \quad \text{(2)} \]
\[ c) \quad \text{HCO}_3^- + \text{H}^+ \rightarrow \text{H}_2\text{O} + \text{CO}_2 \quad \text{(3)} \]

The titration of the water against standard acid using phenolphthalein end point shows completion of reactions (1) and (2) only. This amount of acid used thus corresponds to hydroxide and half of the normal carbonate present. The titration of the water sample against standard acid to methyl orange end point marks the completion of reactions (1), (2) and (3) The amount of acid used for phenolphthalein end point (P) corresponds to half of normal carbonate and hydroxide ions. While the total amount of acid used for methyl orange end point (M) represents the total alkalinity (due to hydroxide, bicarbonate & carbonate ions)

The possible combinations of ions causing alkalinity in water are,

a) hydroxides only, carbonate only, bicarbonate only or
b) (hydroxides & carbonates), (carbonates & bicarbonates) together.

Note: The possibility of hydroxide and bicarbonate ions together is not possible since the combine together to form carbonate ions \( \text{HO}^- + \text{HCO}_3^- \rightarrow \text{CO}_3^{2-} + \text{H}_2\text{O} \)

As hydroxides & bicarbonate cannot exist together, alkalinity of water is due to (hydroxides & carbonates), (carbonates & bicarbonates) together.

Procedure:

Pipette out 25ml of the given water sample into 250ml clean conical flask. Add 2 to 3 drops of methyl orange indicator. Titrate the solution in the conical flask against standard HCl. The colour of the solution changes from yellow to red at the end point. Note the reading. Repeat the same experiment till the concordant values are obtained

RESULT: The total alkalinity of given water sample = ----------- ppm of CaCO₃
Observation:

Normality of HCL = 0.1N

Volume of sample taken for each titration, V = 25 ml.

Burette : Std. HCl
Conical Flask : 25 ml water sample
Indicator : 2-3 drops of Methyl orange
Colour Change : Yellow to red

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Volume of standard HCL used = -------ml

1000 Cm$^3$ of 1N HCL = 50 gm of calcium carbonate

1ml of 1N HCL = 50mg of calcium carbonate

-------- Cm$^3$ of ------ N HCL solution= 50 x ------ x------mg of Calcium carbonate

= --------------mg of Calcium carbonate

25 Cm$^3$ of waste water sample contains   ----------- mg of Calcium carbonate

1000 Cm$^3$ of water sample contains = \[
\frac{1000 \times \text{-----}}{25}
\]

= --------------mg/L of CaCO$_3$

Result: Total alkalinity of the water sample = -----------ppm of CaCO$_3$
DETERMINATION OF pKa OF A WEAK ACID USING pH METER

1. How is pKa of a weak acid evaluated from the graph?
   pH at half equivalence point gives the pKa of a weak acid

2. What is a weak acid?
   Weak acids are weak electrolytes, which ionize incompletely in aqueous solution.

3. Give an example for weak acid?
   Acetic acid, Oxalic acid

4. What is pKa of a weak acid?
   pKa is defined as, negative logarithm to the base 10 of dissociation constant of weak acid. i.e.,
   \[ pKa = - \log_{10}Ka \]
   Where Ka is the dissociation constant of the weak acid.

5. What is meant by pH ?
   pH is defined as negative logarithm to base 10 of hydrogen ion concentration. \( pH = - \log_{10}[H^+] \)

6. What is the modern definition (IUPAC) of pH?
   The difference in pH between two solutions S (std) and X (unknown) at the same temperature
   with the same reference electrode and with hydrogen electrodes at the same hydrogen pressure
   is given by
   \[ pH (X) - pH (S) = \frac{E_X - E_S}{2.3026 RT / F} \]

7. Why glass electrode is called an ion selective electrode?
   Because it is able to respond to certain specific ions (H\(^+\) ions) only and develop a potential
   while ignoring the other ions in a solution.

8. How is the measurement of pH made?
   The measurement of pH is made by determining the emf of the cell containing glass electrode
   and the calomel electrode immersed in the test solution. The emf of the cell is expressed by
   the equation \( E = K + 0.0591 \) pH, where K is a constant.

9. How are pH and pKa related?
   According to Henderson-Hasselbalch equation, \( pH = pKa + \log_{10} \frac{[salt]}{[acid]} \)

10. Why pH at half equivalence point is equal to pKa?
    At half equivalence point concentration of salt = concentration of acid, Therefore \( pH = pKa \)

11. How are pKa and strength of a weak acid related?
    Higher the pKa, lower will be the strength of the weak acid.

12. What are the electrodes used in the measurement of pH and determination of pKa?
    Glass electrode (indicator electrode) and calomel electrode (reference electrode).
    In this case, glass electrode acts as anode & calomel acts as cathode.

13. Why is pH increases suddenly after the equivalence point?
    At the equivalence point, all the weak acid has been neutralized by the base. Afterwards the
    concentration of hydroxyl ions increases. So pH increases suddenly.
1. What is single electrode potential?
The potential that is developed when an element is in contact with a solution containing its
own ions is called single electrode potential

2. What is standard electrode potential?
The potential that is developed when an element is in contact with a solution containing its
own ions in 1M concentration at 298 k is called standard electrode potential

3. What is meant by emf?
EMF is potential difference required to drive current across the electrodes.
\[ \text{EMF} = E_{\text{cathode}} - E_{\text{anode}} \]

4. What is potentiometer?
It is a device or circuit used for measuring the emf between the two electrodes

5. What is potentiometric titration?
It is the measurement of emf to determine the concentration of ionic species in solution is
called potentiometry

6. What is the principle of potentiometric titration?
The measurement of the emf between two electrodes (indicator and reference) and to
determine the equivalence point of redox reaction

7. Which are the electrodes used in potentiometric electrode?
Platinum electrode as indicator electrode (anode) and calomel electrode as reference
electrode (cathode)

8. What is determining factor in the oxidation-reduction reaction?
The determining factor is the ratio of the concentrations of the oxidized and reduced
forms.

9. What is indicator electrode?
The indicator electrode is a electrode whose potential is dependent upon the
concentration of the ion to be determined.

10. What is the reaction occurring between FAS and potassium dichromate?
\[ 6\text{FeSO}_4 + 7\text{H}_2\text{SO}_4 + 2\text{K}_2\text{Cr}_2\text{O}_7 \rightarrow 3\text{Fe}_2(\text{SO}_4)_3 + 2\text{K}_2\text{SO}_4 + \text{Cr}_2(\text{SO}_4)_3 + 7\text{H}_2\text{O} \]
Acidified potassium dichromate oxidizes ferrous sulphate to ferric sulphate and itself gets
reduced to chromic sulphate

11. Why sulphuric acid is added to FAS solution during emf measurement?
In presence of sulphuric acid, potassium dichromate oxidizes ferrous ion(Fe^{2+}) to
ferric ion (Fe^{3+})

12. Why the colour of the solution changes to green on adding potassium dichromate?
Due to formation of green coloured chromic sulphate.

13. Why emf rapidly change at the equivalence point?
At the equivalence point, emf depends on concentration of both ferric ion and
dichromate ion. Therefore emf rapidly change at the equivalence point.
14. What are the advantages of potentiometric titrations?
   i) Turbid, fluorescent, opaque or coloured solutions can be titrated
   ii) Mixture of solutions or very dilute solutions can be titrated.
   iii) The results are more accurate

**COLORIMETRIC DETERMINATION OF COPPER**

1. What is the basic principle of colorimetric measurements?
   It consists of comparing under well defined conditions, the colour produced by the substances
   in amount with the same colour produced by a known amount of material being determined.

2. What is colorimetry?
   Chemical analysis through measurements of absorption of light radiation in the visible region
   of the spectrum (400-760nm) with respect to a known concentration of the substance is known
   as colorimetry.

3. What forms the basis for colorimetric determination?
   The variation of colour of a system with change in concentration of some component forms the
   basis for the colorimetric determination.

4. What is photoelectric colorimeter?
   It is an electrical instrument, which measures the amount of light absorbed using a photocell.

5. What are filters? Why are they used?
   The filter consists of either thin film of gelatin containing different dyes or of coloured glass.
   The filters are used in colorimeter for selecting any desired spectral region.

6. What is wavelength?
   The distance between any two successive peaks or troughs of waves is called wave length. It
   is represented by \( \lambda \).

7. What is wave number?
   It is reciprocal of wavelength. Wave number = \( 1/\lambda \) = Frequency / Velocity of light.

8. What is frequency?
   It is the number of waves passing through a point per second. It is represented by \( \nu \).

   The intensity of a beam of monochromatic light decrease exponentially as the concentration of
   the absorbing substance increases arithmetically.

10. State Lambert’s law.
    When a monochromatic light passes through a transparent medium, the rate of decrease in
    intensity with the thickness of the medium is proportional to the intensity of the light. or The
    intensity of the emitted light decreases exponentially as the thickness of the absorbing medium
    increases arithmetically.

    The amount of light absorbed is directly proportional to the concentration of the solution.
    \[ A = \log I_0 / I_0 = \varepsilon cl \] \( (C = \text{Molar extinction coefficient}, \ c = \text{concentration}, \ l = \text{path length}) \)
    Therefore, \( A \alpha C \)
12. What is calibration curve?
It is the plot of absorbance or Optical Density against concentration of solutions. For solutions obeying Beer’s law, this is a straight line.

13. What is meant by transmittance?
It is the ratio of amount of light transmitted ($I_t$) to the total amount of light absorbed ($I_0$)
$$T = \frac{I_t}{I_0}$$
Absorbance $= \frac{1}{T} = \frac{I_0}{I_t}$

14. What is absorbance or optical density?
Reciprocal of transmittance, $I_0/I_t$ is called absorbance or optical density
$$A = \frac{\log I_0}{I_t} = \log \frac{I_0}{I_t} = -\log T$$

15. How is optical density is related to the concentration of the substance?
The optical density is directly proportional to the concentration of the substance

16. What is a stock solution?
A stock solution is a standard solution in which a known amount of solute dissolved in a known volume of solvent.

17. Can different nessler’s tubes be used during OD measurements of solutions?
No, because absorbance depends on the path length of the solution.

18. Mention a few advantages of photoelectric colourimetric determinations.
i) A colourimetric method will often give more accurate results at low concentrations than the corresponding titrimetric or gravimetric procedure.
ii) A colorimetric method may frequently be applied where no satisfactory gravimetric or titrimetric procedure exits i.e. for certain biological substance.

19. Why are different volumes of solution taken in the flask?
Different volumes of solutions are taken to prepare standard solutions of different concentration, which are used to plot a calibration curve.

20. What is blank solution?
A blank solution is identical in all respect to the test solution except for the absence of test solute.

21. Why is a blank solution used in colorimetric estimation?
To nullify the absorbance caused due to the colouring impurities present in the reagents.

22. Name the complex obtained when ammonia is treated with copper sulphate solution
Cuprammonium sulphate, $[\text{Cu} (\text{NH}_3)_4]\text{SO}_4$ a dark blue colour complex
23. Why is ammonia added? Why is same amount of ammonia added?
   Ammonia is added to get cuprammonium sulphate \([\text{Cu(NH}_3\text{)}_4]\text{SO}_4\) a dark blue complex. Same amount of ammonia is added to nullify the absorbance due to any colouring impurities present in ammonia.

24. Why is estimation of copper done at 620 nm wavelength?
   The estimation of copper is carried at 620 nm wavelength because the complex shows maximum absorbance at 620 nm.

25. Mention a few important criteria for a satisfactory colorimetric analysis.
   i) In order to obey Beer-Lambert’s law, the solute must not undergo salvation, association, dissociation, hydrolysis or polymerization in the solvent used.
   ii) The colour produced should be sufficiently stable to permit an accurate reading to be taken.
   iii) Clear solutions free from traces of precipitate or foreign substances in either blank or standard test solution should be used.

**CONDUCTOMETRIC ESTIMATION OF ACID MIXTRE USING STD. NaOH SOLUTION.**

1. What is conductivity of an electrolyte solution?
   Is a measure of its ability to conduct electricity?

2. What is conductance?
   Reciprocal of resistance is called conductance.

3. What is a cell?
   A device, which produces an EMF and delivers electric current as the results of a chemical reaction.

4. What factors determine the conductance of a solution?
   Two factors determining the conductance of a solution are:
   1. Mobility of ions (Higher the mobility, higher is the conductance) and
   2. Number of ions (More the number of ions, more is the conductance).

5. What is cell constant?
   Is a function of the electrode areas, the distance between the electrodes and the electrical field pattern between the electrodes. Cell constant is defined as the ratio of the distance between the electrodes, \(d\), to the electrode area, \(A\).

6. What is the unit of cell constant?
   Commonly, expressed in \(\text{cm}^{-1}\),
7. State Ohm’s law.
   Current, I (ampere), flowing in a conductor is directly proportional to the applied
   electromotive force, E (volt) and inversely proportional to the resistance R (ohm) of the
   conductor. \[ I = \frac{E}{R} \]

8. What is the unit of conductance?
   Ohm \(^{-1}\) m \(^{-1}\) or Sm \(^{-1}\) (S- siemens).

9. Mentions the different types of conductance.
   i) Specific conductance.
   ii) Equivalent conductance
   iii) Molar conductivity.

10. Which of the above conductivity is measured during the conductometric titration?
    Specific conductance.

11. What is specific conductivity?
    Conductivity of a solution between 1cm\(^2\) area and kept 1 cm apart.

12. What is equivalent conductance?
    Conductivity of a solution, which contains 1g equivalent of solute, when placed between
    2 electrodes of 1cm apart. \( \lambda = kV \).

13. What is molar conductivity?
    Conductivity of the solution, which contains 1g molecular weight of a substance, when
    placed between two electrodes of 1 cm apart, \( \mu = kV \).

14. What is the principle involved in conductometric titration?
    There is a sudden change in conductance of a solution near the end point. Hence the end
    point is determined graphically by plotting conductance against the titer values.
    Measuring resistance and the dimension of conductivity cell (Ohm law) and plotting the
    titration curve (conductance-as reciprocal of resistance against the volume of titrant) is the
    principle.

15. Explain the variation in conductivity at the end point.
    In the case of NaOH and HCl experiment, addition of NaOH to HCl decreases the
    conductance because of the replacement of high conducting mobile H\(^+\) ions (Conductivity :
    350 Ohm\(^{-1}\)m\(^{-1}\)) by the less mobile Na\(^+\) ions (Conductivity : 50 Ohm\(^{-1}\)m\(^{-1}\)). Trend
    continues till all the hydrogen ions are replaced and end point is reached. Further addition
    of NaOH, increases the conductance because of the presence of free OH\(^-\) ions
    (Conductivity : 198 Ohm\(^{-1}\)m\(^{-1}\)).

16. Why are the slopes of curves before and after neutralization point not exactly the same?
    Before the neutralization point, the conductivity is mainly due to the highly mobile H\(^+\)
    ions and after the end point, it is mainly due to OH\(^-\) ions, which are less mobile compared
    to H\(^+\) ions. As a result, magnitude of decrease in conductivity is more before the
    neutralization point compared to that after the neutralization point.

17. How is the accuracy of the method determined?
    Accuracy of the method is greater when the angle of intersection is more acute and more
    nearly the end points of the graph on a straight line.
18. What are the advantages of conductometric titration over visual potentiometric titrations?
   i). Accurate for both dilute and concentrated solutions.
   ii). Can be employed for colored solutions.
   iii). Very weak acids such as boric, phenol (cannot be titrated potentiometrically) can be titrated.
   iv). Mixture of acids can be titrated more accurately.
DETERMINATION OF VISCOSITY COEFFICIENT OF A GIVEN LIQUID USING OSTWALD’S VISCOMETER

1. What is viscosity?
   Viscoisty arises due to internal friction between moving layers of molecules. A moving layer exerts a drag or friction on its nearest moving layer backward. This property of a liquid by which it retards or opposes motion between the layer is called viscosity.

2. What is viscosity co-efficient of a liquid?
   The viscosity coefficient of a liquid is defined as the tangential force per unit urea required to maintain a unit velocity gradient between any two successive layers of a liquid situated unit distance apart.

3. What is density of liquid?
   The density of a liquid is the mass of its unit volume.

4. The density of a substance is expressed relative to what?
   The density of a substance is expressed relative to that of water at 4°C.

5. What is specific gravity?
   Specific gravity or relative density is the weight of a given liquid divided by the weight of an equal volume of water at the same temperature.

6. How are specific gravity and density of liquid related?
   Density of liquid = specific gravity of the liquid x Density of water at the same temperature.

7. What is the SI unit of viscosity coefficient?
   Pascal second (1 ps = 1 kg m\(^{-1}\) s\(^{-1}\)).

8. What is the unit of viscosity coefficient in cgs system?
   Millipoise.

9. What are the factors that affect the viscosity of a liquid?
   i) Increase in molecular weight results in an increase in viscosity.
   ii) Branched chain compounds have higher viscosity than those involving straight chain compounds.
   iii) The polar compounds are more viscous than the non polar ones. The presence of hydrogen bonds cause the viscosity to increase.
   iv) The viscosity of liquids increases by the presence of lyophilic colloids and suspended impurities.
   v) Temperature has marked influence on the viscosity of a liquid (about 2% per degree).

10. What is the law based on the viscous flow of liquids through capillary tubes?
    Poiseuille law
    \[
    \eta = \frac{\Pi Pr^4t}{8Vl}
    \]
    Where P = hgd
    V = volume of the liquid, r = radius of the tube, l = length of the tube,
    \(\rho\) = pressure difference between two ends of the tube,
    \(\eta\) = the coefficient of viscosity of the liquid,
    t = time required for the volume.
11. How does viscosity vary with temperature?
   The viscosity of a liquid usually decreases with rise in temperature.

12. Why should the viscometer be dried before the measurements are done?
   The viscometer should be dried to avoid the formation of emulsion, which changes the rate of flow of the liquid.

13. Why is acetone used for cleaning viscometer?
   Acetone is volatile organic liquid. To dry the viscometer quickly, it is rinsed with acetone.

14. Why is viscometer not rinsed with the given liquid or water?
   If the viscometer is rinsed with given liquid or water before measuring the flow time, the volume taken will be more than a definite known volume.

15. Why do you require laboratory temperature for viscosity determination?
   Because the physical constants like density and viscosity of a liquid vary with temperature.

16. How is the viscosity of a liquid related to its mobility?
   Viscosity of a liquid is inversely proportional to its mobility.

17. What is fluidity of a liquid?
   Fluidity of liquid is the reciprocal of viscosity co-efficient. It is a measure of the ease with which the liquid flows.
   Fluidity (\(\Theta\)) = \(1/\eta\), \(\eta\) = the viscosity co-efficient.
FLAME PHOTOOMETRIC ESTIMATION OF SODIUM IN THE GIVEN SAMPLE OF WATER.

1. What is flame photometry?
   Flame photometry is an atomic emission method for the detection of metal salts like Sodium, Potassium etc, by measuring the flame emission of respective metal salt solutions.

2. What are alkali and alkaline earth metals?
   The elements of first groups, are called alkali metals (Li, Na, K, Rb and Cs). Elements of second group are called alkaline earth metals (Be, Mg, Ca, Sr and Ba).

3. What are the various events that occur, when a solution containing metal atom ion Atomized through a flame?
   Sequence of events taking place are:
   a. Aspiration of liquid sample into a flame.
   b. Formation and evaporation of liquid droplets resulting in the formation of residue.
   c. Decomposition of residue into neutral atoms.
   d. Excitation of atoms and emission of radiation from atoms.
   e. Measurement of wavelength and intensity of emitted radiation by flame photometer.

4. What are different components of a flame photometer?

   ![Schematic diagram showing the layout of various components of a flame photometer](image)

   An atomizer, the burner system and an optical system, a photocell, amplifier and recorder.

5. Why the analysis of Sodium and Potassium is advantageous in flame photometry?
   1. Is fast, simple and reliable.
   2. Sensitivity in the ppm and sub-ppm range.
   3. Flame photometer is portable and its ease of operation.

6. What is the role of filter in flame photometry?
   Permits only the radiation characteristics of the element under investigation.

7. What are the errors that occur in flame photometry?
   An extra molecular entity formed lowers the efficiency. The low temperature renders this method susceptible to certain disadvantages, most of them related to interference and the stability (or lack thereof) of the flame and aspiration conditions.
8. What are the factors that influence this experiment?
   Fuel, oxidant flow rates, purity aspiration rates, solution viscosity, contaminants in the
   samples etc affect the experiment.

9. Name the fuel and oxidant used in this experiment?
   LPG-fuel and air as oxidant.

10. Why blank test is essential?
    A solution of blank is run for setting zero emission.
DETERMINATION OF PERCENTAGE OF COPPER IN BRASS USING STANDARD SODIUM THIOSULPHATE SOLUTION.

1. What is Brass?
   Brass is an alloy

2. What is an alloy?
   An alloy is a homogeneous mixture of two or more metals

3. What are the constituents of brass?
   Brass is an alloy of copper (50-90%), zinc (20-40%), small quantities of tin, lead and iron.

4. How is brass solution prepared?
   Brass solution is prepared by dissolving the brass foil in 1:1 nitric acid.

5. What is the purpose of adding urea to the brass solution?
   Urea is added to destroy excess of nitrous acid and oxides of nitrogen which interfere with the determination. If they are not destroyed they also oxidize KI to I\(_2\) which should have been done by Cu\(^{+2}\) ions

   \[
   2\text{NO} + \text{O}_2 \rightleftharpoons 2\text{NO}_2 \\
   2\text{NO}_2 + \text{H}_2\text{O} \rightleftharpoons \text{HNO}_2 + \text{HNO}_3 \\
   
   \]

   \[
   2\text{HNO}_2 + \text{H}_2\text{N} \rightleftharpoons 2\text{N}_2 + \text{CO}_2 + 3\text{H}_2\text{O} \\
   \]

6. Why ammonium hydroxide is added to the brass solution?
   Ammonium hydroxide is added to neutralize the mineral acid (HNO\(_3\) & HNO\(_2\))

7. What is the need to neutralize the mineral acid?
   The mineral acid HNO\(_3\) is a strong oxidizing agent. It oxidize KI to I\(_2\) therefore the amount of iodine liberated does not corresponds to the exact concentration of Cu\(^{+2}\) ions.

8. What is the bluish white precipitate formed after adding ammonia solution?
   Cupric hydroxide

9. Why acetic acid is added?
   Acetic acid is added to neutralize the excess of ammonium hydroxide and to make the solution slightly acidic.

10. Why is acidic medium necessary?
    The oxidation of KI to I\(_2\) takes place in acidic medium.

11. Why is KI added to brass solution although copper in brass is determined?
    Cupric ions do not react with sodium thiosulphate solution. Cupric ions oxidize KI and iodine is liberated. The amount of iodine is liberated is equal to the amount of cupric ions present in the solution.
12. Although copper ions are blue in colour in the beginning become colorless after the end point? why?
   At the beginning of the titration Cu²⁺ ions present (blue) which are reduced to Cu⁺ ions (colorless) state.

13. Why do you get blue color when starch indicator is added?
   Triiodide in the solution reacts with the amylose of starch gives a blue color.

14. How is liberated iodine estimated?
   The amount of iodine liberated is estimated by titrating it against standard sodium thiosulphate solution using starch as an indicator.

15. What is the reaction that occurs between iodine & sodium thiosulphate?
   \[ 2Na_2S_2O_3 + I_2 \rightarrow Na_2S_4O_6 + 2NaI \]

16. Why is starch indicator added towards end point?
   If the intensity of iodine is more, the starch indicator forms a stable water insoluble complex with iodine. As a result the volume of sodium thiosulphate will be more than expected.

17. What is the white precipitate produced at the end point?
   Cuprous iodide
PREPARATION OF STANDARD POTASSIUM DICHROMATE SOLUTION AND DETERMINATION OF IRON IN THE GIVEN SAMPLE OF HAEMATITE ORE SOLUTION. (External indicator method)

1. What is an ore?
   An ore is mineral, from which a metal can be extracted economically & conveniently.

2. What is a mineral?
   A mineral is a naturally occurring metallic compound.

3. What are the main constituents of haematite ore?
   Ferric oxide (Fe₂O₃)

4. Give the other forms of iron ore?
   Magnetite (Fe₃O₄), Siderite (FeCO₃), Iron pyrite (FeS₂)

5. Why HCl is added?
   HCl is added to convert Fe₂O₃ to FeCl₃, as Fe₂O₃ does not react with K₂Cr₂O₇.

6. What is the role of stannous chloride?
   Stannous chloride reduces Ferric ion to ferrous ion and a slight excess is added to ensure complete reduction. 2FeCl₃ + SnCl₂ → 2 FeCl₂ + SnCl₄

7. Why is mercuric chloride added?
   To remove excess of stannous chloride. Mercuric chloride reacts with stannous chloride to form a silky white precipitate of mercurous chloride.(Calomel)
   SnCl₂ + 2HgCl₂ → Hg₂Cl₂ + SnCl₄

8. What happen when excess of stannous chloride is not removed?
   The ferric ions formed during the course of the titration get reduced to ferrous ions. As a result, the volume of potassium dichromate consumed will be more.

9. Which is the indicator used?
   K₃[Fe(CN)₆], Potassium ferri cyanide as external indicator.

10. Why potassium ferri cyanide cannot be used as an internal indicator?
    Because it combines irreversibly with ferrous ion to form a deep blue ferrous ferri cyanide complex (Turnbull’s blue). These ferrous ions involved in complex formation are not available for reaction with potassium dichromate. Moreover end point cannot be detected.
    3 Fe²⁺ + 2 K₃[Fe(CN)₆] → Fe₃[Fe(CN)₆]₂ + 6K⁺

11. Why potassium Ferro cyanide cannot be used as an indicator in the estimation of Fe
    Potassium Ferro cyanide cannot be used as an indicator because Ferro cyanide does not react with ferrous ion.

12. Why the colour of the indicator drop remains same at the end point?
    At the end point there are no more ferrous ions available to react with the indicator, as they oxidized to ferric ions by the time the end point is reached.

13. What is the reaction that occurs during the titration?
Acidified potassium dichromate oxidizes ferrous iron present in the haematite solution to ferric iron and itself gets reduced to chromic ion.

\[
6\text{Fe}^{2+} + \text{Cr}_2\text{O}_7^{2-} + 14\ \text{H}^+ \rightarrow 6\ \text{Fe}^{3+} + 2\text{Cr}^{3+} + 7\text{H}_2\text{O}
\]

14. **Can Fe be determined using internal indicators?**
   Yes, Fe can be determined using internal indicators.

15. **Name the internal indicator used for iron determination?**
   Ex: a) Diphenyl amine b) N-phenylanthranilic acid
PREPARATION OF STANDARD EDTA SOLUTION AND DETERMINATION OF TOTAL HARDNESS OF A SAMPLE OF WATER

1. **Why is ammonia ammonium chloride buffer added?**
   Ammonia ammonium chloride is added to maintain a pH of 10. Otherwise pH decreases as H\(^+\) ions are released due to the substitution of metal ions for H\(^+\) ions in EDTA.

2. **What is hard water?**
   Hard waters are a kind of water that requires considerable amounts of soap to produce foam and lather.

3. **What is total hardness of water?**
   The sum of both temporary and permanent hardness is called total hardness of water.

4. **How are the waters classified based on the degree of hardness?**
   - Soft water 0 to 75 mg / litre
   - Moderately hard water 75 to 150 mg / litre
   - Hard water 150 to 300 mg / litre
   - Very hard water > 300 mg / litre

5. **How is hardness of water caused?**
   Hardness of water caused by divalent metallic cations in association with anions such as HCO\(_3\)\(^-\), SO\(_4\)\(^{2-}\), Cl\(^-\), SiO\(_3\)\(^{2-}\) etc. the principal hardness causing cations are calcium and magnesium.

6. **What is the difference between temporary and permanent hardness?**
   Temporary hardness is due to unstable bicarbonates of Ca and Mg, while permanent hardness is due to more stable Cl\(^-\) and SO\(_4\)\(^{2-}\) of Ca and Mg

7. **How temporary hardness is be removed?**
   The temporary hardness of water can be removed by boiling water during which bicarbonates decomposes to give carbonates.

8. **How do you express the total hardness of water?**
   It is expressed as parts per million of CaCO\(_3\).

9. **What is EDTA?**
   Ethylene diamine tetra acetic acid.

10. **Write a structural formula of EDTA.**
    ![Structure of EDTA]

11. **How many replaceable hydrogen atoms are present in disodium salt of EDTA?**
    Two.

12. **Why is disodium salt of EDTA preferred to EDTA?**
13. **Why is ammonia solution added while preparing EDTA solution?**
   Because to increase the rate of dissolution of EDTA salt.

14. **What is buffer capacity?**
   The capacity of a solution to resist the change in pH.

15. **Give an example of acidic buffer?**
    CH₃COOH + CH₃COONa

16. **Give an example of basic buffer?**
    NH₄OH + NH₄Cl

17. **What is reserve alkalinity?**
    Buffer solutions are considered to possess reserve acidity as well as alkalinity.
    According to Henderson-Hasselbalch equation, pH = pKb + log₁₀ [salt] / [base],
    At half equivalence point [ Concentration of salt = Concentration of base],
    Therefore pH = pKb. Similarly, for reserve acidity for acidic buffer.

18. **What is buffer solution?**
    The solution which resists change in its pH value even after adding small amounts of an acid or base to it.

19. **What is the chemical name of EBT?**
    Sodium 4-[(1-hydroxynaphthalen-2-yl-hydrazinylidene]-7-nitro-3-oxo-Y-naphthalene-1-sulfonate.

20. **Why is EBT shows wine red at the beginning and blue colour at the end?**
    EBT is blue in colour, which is added to hard water with a pH of 10; it combines with a few of the calcium and magnesium ions to form a weak complex which is wine red in colour as shown in the equation.

    \[
    M^{2+} + \text{EBT} \rightarrow M\text{-EBT} \quad \text{(Wine red complex)}
    \]

    \[
    M^{2+} + \text{EDTA} \rightarrow M\text{-EDTA} + \text{EBT (Blue)} \quad \text{(Stable complex) (protonated)}
    \]

    This action frees the EBT from the complex (M-EBT), and the wine red color changes to a blue colour at the end of titration.

21. **Titrations involving EDTA carried out slowly towards the end point. Justify?**
    Because the rate of formation of the metal complex of EDTA is very slow.
22. **What is the application of hardness data in environmental engineering practice?**
   i) Hardness of water is an important consideration in determining the suitability of a water for domestic and industrial uses.
   ii) Determination of hardness serves as a basis for routine control of softening processes.
PREPARATION OF STANDARD EDTA SOLUTION AND DETERMINATION OF PERCENTAGE OF CALCIUM OXIDE IN THE GIVEN SAMPLE OF CEMENT SOLUTION. (Rapid EDTA Method)

1. What is cement?
   Cement is a lime based building material used to bind together coarse aggregates.

   CaO = 60-66%, SiO₂ = 17-25%, Al₂O₃ = 3-8%, Fe₂O₃ = 2-6%, MgO = 0.1 – 5.5%, Na₂O & K₂O = 0.5 – 1.5% and SO₃ = 1-3%

3. How is cement solution prepared?
   Accurately weighed amount of cement is warmed with moderately conc. HCL till cement dissolves insoluble silica is filtered off & filtrate is the cement solution

4. Why is the titration called rapid EDTA method?
   The titration is called rapid EDTA method because calcium ions in cement solution are estimated directly & quickly by using standard EDTA solution without removing the other metal ions.

5. What is the role of NaOH in this titration?
   NaOH reacts with magnesium ion & precipitates as magnesium hydroxide from the cement solution.  Mg²⁺ + 2NaOH → Mg(OH)₂ + 2 Na⁺

6. What are the constituents of cement?
   Oxides of calcium, magnesium, iron, aluminum and silicon.

7. What is the main constituent of cement?
   Calcium oxide.

8. What is the role of glycerol?
   Glycerol is added to get sharp end point.

9. What is the role of NaOH?
   NaOH is added to mask the magnesium ions.

10. What is the function of diethyleamine?
    Diethyleamine is added to maintain a pH of about 12.5.

11. Why is EBT indicator cannot be used in this experiment?
    Because it forms a weak complex with calcium ions at pH range of 12-14.
12. Which is the indicator used in the determination of CaO in cement solution?
Patton and reeder’s reagent (Calconcarboxylic acid; IUPAC name: Naphthalenecarboxylicacid,3-hydroxy-4-[(2-hydroxy-4-sulfo-1-naphthalenyl)azo]-;3-hydroxy-4-[(2-hydroxy-4-sulfo-1-naphthalenyl)azo]-2-Naphthalenecarboxylicacid).
DETERMINATION OF CHEMICAL OXYGEN DEMAND (COD) OF THE INDUSTRIAL 
WASTE WATER SAMPLE

1. What is sewage?
   Sewage is commonly a cloudy dilute aqueous solution containing human & household 
   waste, industrial waste, ground waste, street washings. Sewage contains organic and 
inorganic matters in dissolved, suspended & colloidal states.

2. What is meant by industrial sewage?
   The waste water coming out of industries such as chemical plant, fertilizer industries, 
   leather tanneries, sugar and paper industries, breweries, textile mills, oil refineries, 
   pharmaceutical units is called industrial sewage.

3. What is COD?
   It is amount of oxygen required for the complete chemical oxidation of both organic and 
inorganic matter present in the sample of water by a strong chemical oxidizing agent such as 
acidified potassium dichromate.

4. What are the compounds present in industrial waste water sample?
   Waste water contains organic impurities which include straight chain aliphatic compounds 
aromatic hydrocarbons, straight chain alcohols, acids and other oxidisable materials.

5. What is BOD?
   It is the amount of oxygen required for the complete oxidation of organic matter present in 
liter of waste water over period of 5 days at 20° C.

6. Why COD greater than BOD value?
   Because in COD both organic & inorganic matters gets oxidized & in BOD only organic 
matters gets oxidized.

7. What are end products of oxidation?
   CO₂ & H₂O.

8. Why H₂SO₄ is added to conical flask?
   Because Potassium dichromate acts as an oxidizing agent in presence of H₂SO₄.

9. What is the unit of COD?
   mg/litr of O₂ or mg/dm³ of O₂ or ppm of O₂.

10. Name the oxidizing agent used in the COD test?
    Potassium di chrome.

11. Explain the colour change during the titration.
    Ferroin indicator is red in colour in the reduced form with the composition, [Fe(O-
    Phen)₃]²⁺(ferroin), When ferroin is added to conical flask containing waste water & potassium 
dichromate. The indicator gets oxidized by potassium dichromate (oxidizing agent) to form 
[Fe(O-Phen)₃]³⁺(ferrin), which is bluish green. This solution is titrated with FAS taken in the 
burette (reducing agent) which reduces ferrin to Ferroin which is in red colour which is the end 
point of titration.
12. **What is back titration?**
   Back titration is a technique used to find the concentration of the reactant by reacting with an excess volume of reactant of known concentration.

13. **Differentiate between blank & back titration. Why blank value is higher than back value?**
   Blank titration is done using distilled water instead of test solution (waste water) and back titration is done using waste water as one of the solvent. Blank titration involves no oxidation of organic matter.

14. **What general groups of organic compounds are not oxidized in the COD test?**
   Aromatic hydrocarbons and pyridine are not oxidized in COD test.

15. **What is the role of silver sulphate?**
   Silver sulphate acts as a catalyst in the oxidation of straight chain aliphatic hydrocarbons and acetic acid. Oxidation is effective in presence of silver ions.

16. **What is the role of mercuric sulphate?**
   Chloride ions normally present in high concentration in waste water undergo oxidation in COD test and cause erroneously high results. Mercuric ions of mercuric sulphate bind the halide ions present in waste water to form poorly ionized mercuric chloride and prevent the precipitation of silver halide by making halide ions unavailable.

17. **If nitrites are present as impurities, what is the reagent used?**
   Sulphamic acid (amido sulphonic acid, H$_3$NSO$_3$)

18. **What are the products formed after COD analysis?**
   During COD analysis organic matter is completely oxidized to carbon dioxide and water by acidified potassium dichromate solution.

19. **Why is sulphuric acid added during the preparation of std. FAS solution?**
   Sulphuric acid is added to prevent the hydrolysis of ferrous sulphate into ferrous hydroxide.

20. **What is the composition of ferroin?**
   Ferroin is ferrous 1, 10-Phenanthroline sulphate.
21. **Why blank titration is necessary?**

Because the COD measures the oxygen demand in the sample of water, it is important that no outside organic material be accidental added to the sample to be measured. To control for this, so called blank sample is required.

22. **Mention a few applications of COD test in environmental engg.?**

   i) The COD test is extensively used in the analysis of industrial wastes
   
   ii) It is particularly valuable in survey designed to determine and control losses to sewer systems
   
   iii) The COD is helpful in indicating toxic conditions and the presence of biologically resistant organic substances.

23. **What is the limitation of COD?**

One of the chief limitations of COD test is inability to differentiate between biologically oxidisable and biologically inert organic matter. Also, it does not provide any evidence of the rate at which the biologically active material would be stabilized under conditions that exits in nature.
DETERMINATION OF TOTAL ALKALINITY OF WATER SAMPLE

1. What is alkalinity?
   It is the capacity to neutralize acid.

   Note: The alkalinity in water is due to hydroxyl only, or carbonate only, or bicarbonate only because they combine instantaneously to form carbonate ions
   \[ \text{OH}^- + \text{HCO}_3^- \rightarrow \text{CO}_3^{2-} + \text{H}_2\text{O} \].
   All three hydroxyl, carbonate, and bicarbonate cannot exist each other.

2. What are the causes for alkalinity?
   Alkalinity is due to the presence of substances in water, which have tendency to increase the concentration of hydroxyl, carbonates and bicarbonates or dissociation of salt in water.

3. Name the compounds which causes alkalinity?
   a) Presence of salts of weak acid which undergo hydrolysis and consume H\(^+\) of water.
   b) Presence of bicarbonates, silicates etc., in water take up H\(^+\) from water. Hence, concentration of OH\(^-\) in water increases and cause alkalinity.
   c) Presence of salts like sodium carbonates, calcium hydroxide, zinc carbonates, calcium bicarbonate, sodium hydroxide etc.,

4. Name the types of alkalinity?
   Phenolphthalein alkalinity- hydroxyl alkalinity, half of carbonate alkalinity.
   Methyl orange alkalinity-bicarbonate alkalinity

5. What are the indicators used in the determination of alkalinity?
   Phenolphthalein and Methyl orange.

6. What is the working pH of phenolphthalein and Methyl orange indication?
   pH = 8.3-10.0 for phenolphthalein; pH=3.1-4.4 for methyl orange.

6. Write the structure and give the colours for phenolphthalein and methyl orange indicators

![Phenolphthalein and Methyl orange indicators]

Yellow color (above pH 4.4)
Red color (below pH 3.1)
Becomes orange (less acidic)
phenolphthalein: pH < 0 (orange),

pH ~ 0–8.2

Structure of phenolphthalein, when pH < 0

7. What is total Alkalinity?
   It is the sum of alkalinity due to hydroxides, alkalinity due to carbonates and alkalinity due to bicarbonates.
COLORIMETRIC DETERMINATION OF COPPER

Draw out 2,4,6,8 & 10 ml of given copper sulphate solution into 5 separate 50 ml volumetric flasks. Add 5ml ammonia to each of them & also into the test solution of unknown concentration. Dilute up to the mark using distilled water & mix well. Measure the absorbance of each of these against blank solution at 620 nm (only ammonia & water). Plot a graph of absorbance against volume of copper sulphate & determine the amount of copper in the test solution.

![Graph](image)

X – vol of test solution

Vol. of CuSO₄ →

FLAME PHOTOMETRIC ESTIMATION OF SODIUM IN THE GIVEN WATER SAMPLE

Draw out 5,10,15,20 & 25 ml of given NaCl solution into 5 separate 50 ml volumetric flasks. Dilute up to the mark with distilled water & mix well. Measure the emission intensity at 589 nm. Plot a graph of emission intensity against volume of NaCl & determine the amount of Na in the test solution.

![Graph](image)

X – vol of test solution

Vol. of NaCl →
CONDUCTOMETRIC ESTIMATION OF ACID MIXTURE USING STD. NaOH SOLUTION.

Pipette out 50ml of acid mixture into a beaker. Immerse the conductivity cell in it & connect it to conductometer and measure the conductance. Add NaOH from the burette in the increments of 0.5 ml and measure the conductance after each addition.

Plot a graph of conductance against volume of NaOH & determine the equivalence point. Calculate the normality & weight of HCl & CH$_3$COOH in the given solution.

DETERMINATION OF VISCOSITY COEFFICIENT OF A GIVEN LIQUID USING OSTWALD’S VISCOMETER

Pipette out 20 ml of given liquid into a wide limb of viscometer and suck the liquid through the other limb. Determine the time flow between 2 fixed points. One above & one below the bulb in the narrow limb of viscometer. Repeat and calculate the average time flow. Pour out the liquid, wash the viscometer with acetone and dry it. Now pipette out 20ml of water into limb and determine the average time flow for water as before. Determine the coefficient of viscosity of the given liquid using the formula.

\[
\eta_L = \frac{\eta_w \times t_L \times d_L}{t_w \times d_w}
\]

\(\eta_w\) = Viscosity coefficient of water
\(\eta_L\) = Viscosity coefficient of liquid
\(d_w\) = density of water
\(d_L\) = density of liquid
DETERMINATION OF pKa OF A WEAK ACID USING pH METER

Pipette out 25ml of the given weak acid into a beaker. Immerse the glass and calomel electrode assembly into it. Connect the electrodes to pH meter and measure the pH. Add NaOH from the burette in increments of 0.5ml and measure the pH after each addition. Plot a graph of ΔpH / ΔV against volume of NaOH and determine equivalence point. Plot another graph of pH against volume of NaOH and determine pKa value of the given weak acid.

\[
\frac{\Delta pH}{\Delta V}
\]

\[
\text{Vol. of NaOH} \rightarrow X
\]

Where X = Equivalence point; X/2 = Half Equivalence point

POTENTIOMETRIC ESTIMATION OF FAS USING STANDARD K₂Cr₂O₇ SOLUTION

Pipette out 25ml of the given FAS solution into a beaker. Add one test tube of dilute sulphuric acid. Immerse the Platinum and calomel electrode assembly into it. Connect the electrodes to potentiometer and measure the potential. Add K₂Cr₂O₇ from the burette in increments of 0.5ml and measure the potential after each addition. Plot a graph of ΔE / ΔV against volume of K₂Cr₂O₇ and determine the equivalence point. From the normality of K₂Cr₂O₇, calculate the normality and weight of FAS present in the given solution.

\[
\frac{\Delta E}{\Delta V}
\]

\[
\text{Vol. of K₂Cr₂O₇} \rightarrow X
\]

Where X = Equivalence point
DETERMINATION OF PERCENTAGE OF COPPER IN BRASS USING STANDARD SODIUM THIOSULPHATE SOLUTION.

Weigh out the given brass sample into a clean Beaker. Add ¼ test tube, con. HNO₃ till brass dissolves completely. Add 1 test tube of distilled water and 1 gm of urea, boil till all brown fumes are expelled. Cool to room temperature. Transfer the solution in to a 250ml volumetric flask and dilute upto the mark with distilled water and mix well.

Burette : Std. Sodium thio sulphate solution
Conical Flask : 25ml Brass solution + NH₄OH drop wise till bluish white ppt. + ¼ test tube dil CH₃COOH + 1 t.t. 10 % KI.
Indicator : Starch - added towards the end point.
Colour Change : Blue to white ppt.

From the volume of Na₂S₂O₃ Consumed, calculate the percentage of copper in the given brass sample.

DETERMINATION OF IRON IN THE GIVEN SAMPLE OF HAEMATITE ORE SOLUTION. (External indicator method)

Part A: Preparation of standard K₂Cr₂O₇ solution
Weigh out the given K₂Cr₂O₇ crystals accurately into a 250ml volumetric flask. Dissolve in distilled water and dilute up to the mark, mix well.

Normality of K₂Cr₂O₇ = \frac{\text{Weight of K}_2\text{Cr}_2\text{O}_7 \text{ taken} \times 4}{\text{Equivalent weight of K}_2\text{Cr}_2\text{O}_7 \text{ (49)}}

Part B: Estimation of Iron in haematite

Burette : Std. K₂Cr₂O₇ Solution
Conical Flask : 25 ml Haematite ore solution + 1/4th test tube of con. HCl, boil + SnCl₂ drop wise till colorless. Cool & add 1/4th test tube HgCl₂
Indicator : Potassium ferricyanide
Color Change : Indicator fails to give any color with the test solution drop

From the volume of K₂Cr₂O₇ consumed, calculate the percentage of iron in the given haematite ore solution.
DETERMINATION OF TOTAL HARDNESS OF A GIVEN SAMPLE OF WATER

Part A: Preparation of standard solution

Weigh out the given disodium salt of EDTA crystals accurately into a 250ml volumetric flask. Add quarter test tube of ammonia. Dissolve in distilled water and dilute up to the mark, mix well.

\[
\text{Molarity of EDTA} = \frac{\text{Weight of EDTA} \times 4}{\text{Molecular weight of EDTA} \times 372}
\]

Part B: Determination of total hardness

Burette : Std. EDTA Solution.
Conical Flask : 25 ml water sample + 3ml Amonia–ammonium chloride buffer solution.
Indicator : Eriochrome black-T.
Colour Change : Wine red to blue.

From the volume of EDTA consumed, calculate the total hardness of the given water sample.

DETERMINATION OF PERCENTAGE OF CALCIUM OXIDE IN THE GIVEN SAMPLE OF CEMENT SOLUTION

Part A: Preparation of standard solution

Weigh out the given disodium salt of EDTA crystals accurately into a 250ml volumetric flask. Add quarter test tube of ammonia. Dissolve in distilled water and dilute up to the mark, mix well.

\[
\text{Molarity of EDTA} = \frac{\text{Weight of EDTA taken} \times 4}{\text{Molecular weight of EDTA} \times 372}
\]

Part B: Determination of % of CaO in cement solution

Burette : Std. EDTA Solution
Conical Flask : 25 ml Cement solution + 5 ml Diethylamine + 5 ml Glycerol + 10 ml NaOH
Indicator : Patton & Reeder’s
Colour Change : Wine red to blue

From the volume of EDTA consumed, calculate the percentage of CaO in the given Cement solution.
DETERMINATION OF CHEMICAL OXYGEN DEMAND (COD) OF THE INDUSTRIAL WASTE WATER SAMPLE

Part A: Preparation of standard FAS solution (Mohr’s salt solution)

Weigh out the given ferrous ammonium sulphate (FAS) crystals accurately into a 250ml volumetric flask. Add 1 t.t. of H$_2$SO$_4$. Dissolve in distilled water and dilute upto the mark, mix well.

\[
\text{Normality of FAS} = \frac{\text{Weight of FAS taken} \times 4}{\text{Equivalent weight of FAS (392)}}
\]

Part B: Determination of COD of waste water sample

Burette : Std. FAS Solution
Conical Flask : 25 ml Waste water + 10ml K$_2$Cr$_2$O$_7$ + 1 t.t. of 1:1 H$_2$SO$_4$ (Boil & Cool)
Indicator : Ferroin
Colour Change : Bluish green to reddish brown.

Perform the blank titration in the same way as above, without taking waste water. From the difference in the titre values, calculate the COD of waste water sample.

DETERMINATION OF TOTAL ALKALINITY OF WATER SAMPLE

Burette : Std. HCl
Conical Flask : 25 ml water sample
Indicator : 2-3 drops of Methyl orange
Colour Change : Yellow to red

From the volume of HCl consumed, calculate the total alkalinity of water sample
Questions

1. Write a brief procedure for the determination of total hardness of water.
2. Write a brief procedure for the determination of Percentage of CaO in cement solution
3. Write a brief procedure for the determination of Percentage of Cu in Brass solution
4. Write a brief procedure for the determination of Percentage of Fe in Haematite solution
5. Write a brief procedure for the determination of COD of waste water.
6. Write a brief procedure for the estimation of FAS potentiometrically.
7. Write a brief procedure for the determination of pKa of Weak acid.
8. Write a brief procedure for the estimation of Cu colorimetrically.
9. Write a brief procedure for the estimation of an acid mixture using std. NaOH
10. Write a brief procedure for the determination of viscosity coefficient of a liquid using Ostwalds viscometer.

Reference Books:

General Instructions for Volumetric Analysis

[Diagram showing the process of volumetric analysis with labels and steps for correct and incorrect做法.

- Correct:
  - Hold the bottle at a right angle.
  - Read the volume at the bottom of the meniscus.

- Incorrect:
  - Hold the bottle at an angle.
  - Read the volume at the top of the meniscus.

Instructions:

1. Hold the bottle at a right angle.
2. Read the volume at the bottom of the meniscus.
3. Align the lower mark of the liquid with the lower edge of the meniscus.
4. Note the volume.

Guidelines:

- Use white paper underneath the bottle to view the volume more clearly.
- Always use a clean and dry bottle.
- Ensure the bottle is at the same temperature as the surrounding environment.
- The temperature of the meniscus should match the temperature of the surrounding environment.

[Additional diagrams showing correct and incorrect methods of pouring and handling reagents.]

- Correct:
  - Pour the solution gently without splashing.
  - Use a spatula or dropper to transfer reagents.

- Incorrect:
  - Pour the solution aggressively, causing splashes and mixing.
  - Use a large, wide-mouthed bottle for transferring reagents.

Tips:

- Use a pipette for precise volume measurements.
- Calibration of pipettes is essential before use.
- Always use appropriate glassware for the solution.
- Clean glassware thoroughly before use.

[Further diagrams showing the correct use of test tubes and flasks, emphasizing the importance of cleanliness and proper handling techniques.]