DIRECTORATE OF DISTANCE & CONTINUING EDUCATIONS

MANONMANIAM SUNDARANAR UNIVERSITY

TIRUNELVELI – 627012

OPEN AND DISTANCE LEARING(ODL) PROGRAMMES

(FOR THOSE WHO JOINED THE PROGRMMES FROM THE ACADEMIC YEAR 2023 – 2024)

B.Sc. PHYSICS COURSE MATERIALS ALLIED CHEMISTRY PRACTICAL – I JECHP1

By

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VOLUMETRIC ANALYSIS

- 1. Estimation of sodium hydroxide using standard sodium carbonate.
- 2. Estimation of hydrochloric acid using standard oxalic acid.
- 3. Estimation of ferrous sulphate using standard Mohr & salt.
- 4. Estimation of oxalic acid using standard ferrous sulphate.
- 5. Estimation of potassium permanganate using standard sodium hydroxide.
- 6. Estimation of magnesium using EDTA.
- 7. Estimation of ferrous ion using diphenyl amine as indicator.

Allied Chemistry Practical for Physical Sciences I

Chemistry is often referred to as the "central science" because it connects physical sciences like physics and mathematics with life sciences and applied sciences such as engineering, medicine, and materials science. While theoretical chemistry lays the foundation for understanding chemical principles, practical chemistry is where the concepts come to life. The laboratory serves as an interactive classroom where students develop the skills necessary to experiment, analyze, and interpret chemical phenomena.

In practical chemistry, every experiment provides an opportunity to engage with the principles of matter, its properties, and its transformations. By conducting experiments, students gain hands-on experience that deepens their understanding of theoretical concepts. Through this practical application, they develop critical thinking, observational skills, and an appreciation for the intricate nature of chemical reactions and processes.

Importance of Practical Chemistry

Practical chemistry plays an essential role in scientific education by:

- 1. Bridging Theory and Practice: Experiments translate abstract theories into observable outcomes, solidifying understanding through application.
- 2. Developing Technical Skills: Students learn to handle laboratory apparatus, prepare solutions, and measure substances with precision.
- 3. Fostering Analytical Thinking: Practical chemistry challenges students to interpret data, troubleshoot errors, and draw meaningful conclusions.
- 4. Encouraging Scientific Curiosity: The laboratory setting sparks curiosity by revealing the wonder of chemical reactions and their real-world applications.
- 5. Building Confidence: Success in the laboratory instills confidence in one's ability to solve problems and apply knowledge in practical scenarios.

Role of Volumetric Analysis

One of the most significant branches of practical chemistry is volumetric analysis. Also known as titrimetric analysis, this technique involves determining the concentration of an unknown solution by reacting it with a standard solution of known concentration. Volumetric analysis is highly versatile and forms the basis for many real-world applications, such as:

- Quality control in industries (e.g., pharmaceuticals and food processing).
- Environmental monitoring (e.g., testing water hardness and pollutant levels).
- Medical diagnostics (e.g., blood glucose level determination).

The experiments in this manual emphasize the importance of volumetric techniques, enabling students to accurately measure and analyze substances. Types of titrations include:

- 1. Acid-Base Titrations: These involve neutralization reactions to determine the concentration of acidic or basic solutions. Indicators like phenolphthalein or methyl orange are used to identify the endpoint.
- 2. Redox Titrations: These involve oxidation-reduction reactions to analyze substances such as ferrous ions, potassium permanganate, and dichromate solutions.
- 3. Complexometric Titrations: These use chelating agents like EDTA to determine metal ion concentrations, commonly applied in water hardness analysis.
- 4. Precipitation Titrations: These involve the formation of a precipitate during the titration, such as the determination of chloride ions using silver nitrate.

Laboratory Safety and Best Practices

Safety is paramount in any laboratory setting. Chemistry experiments often involve handling hazardous substances, glassware, and equipment that require care and attention. Students must adhere to the following safety protocols:

- Personal Protective Equipment (PPE): Always wear lab coats, safety goggles, and gloves.
- Proper Handling of Chemicals: Read labels carefully, and never taste or directly inhale chemicals. Dispose of waste as instructed.
- Fire Safety: Be cautious when working with flammable substances or open flames.
- Equipment Usage: Ensure that apparatus is clean, dry, and used according to instructions.
- Behavior in the Lab: Avoid horseplay, and maintain a clean and organized workspace.

By cultivating these habits, students not only ensure their safety but also learn to work responsibly and professionally in a laboratory environment.

About This Manual

This manual has been designed to provide a structured and comprehensive approach to practical chemistry. It contains a range of experiments that cater to students studying physical sciences and related disciplines. Each experiment includes:

- 1. Aim: A concise statement of the purpose of the experiment.
- 2. Principle: A clear explanation of the underlying chemical reactions and processes.
- 3. Chemicals and Apparatus: A detailed list of the required materials and equipment.
- 4. Procedure: A step-by-step guide to conducting the experiment.
- 5. Observations and Calculations: Tables and formulas for recording and analyzing data.
- 6. Result: A section to summarize the findings.

In addition to technical details, the manual encourages students to reflect on their observations and connect their results to theoretical knowledge. Each experiment has been curated to build foundational skills and promote a deeper understanding of analytical chemistry.

Why Study Practical Chemistry?

Practical chemistry is not limited to academic learning; it has far-reaching implications in various fields:

- Pharmaceuticals: Developing and testing new drugs rely on precise chemical analysis.
- Environmental Science: Monitoring pollutants and ensuring safe water and air quality require chemical testing.
- Materials Science: Innovations in materials, such as corrosion-resistant alloys or biodegradable polymers, depend on a deep understanding of chemical reactions.
- Food Chemistry: Analyzing nutritional content and detecting adulterants ensure food safety and quality.

Key Takeaways for Students

Through this manual, students will:

- Master the art of preparing and standardizing solutions.
- Develop a strong grasp of stoichiometric calculations and reaction mechanisms.
- Learn to interpret experimental data and troubleshoot common errors.
- Appreciate the real-world relevance of chemistry in addressing global challenges like environmental sustainability, healthcare, and industrial innovation.

By the end of this course, students will not only acquire practical skills but also foster a lifelong appreciation for the role of chemistry in shaping our world. Let the experiments inspire curiosity, precision, and a deeper understanding of this fascinating science.

VOLUMETRIC ANALYSIS

1. Estimation of sodium hydroxide using standard sodium carbonate

Aim

To estimate the concentration of sodium hydroxide (NaOH) solution using a standard sodium carbonate (Na2CO₃) solution through volumetric analysis.

Objective

- To determine the concentration of sodium hydroxide solution using a standard sodium carbonate solution.
- To understand the principles of acid-base titration using a primary standard.

Principle

The estimation of sodium hydroxide using standard sodium carbonate is based on the principle of acid-base neutralization, where sodium carbonate (a primary standard) reacts with sodium hydroxide in a titration process. Sodium carbonate is stable, pure, and non-hygroscopic, making it ideal for preparing a standard solution with known concentration. During titration, phenolphthalein is used as an indicator, which is pink in a basic medium and turns colorless at the endpoint, indicating complete neutralization. The reaction involves the interaction of sodium hydroxide with sodium carbonate, and the endpoint is used to calculate the concentration of the sodium hydroxide solution accurately.

$Na_2CO_3 + H_2O \rightarrow 2Na^+ + HCO_3^- + OH^-$

 Na_2CO_3 $(aq) + NaOH$ $(aq) \rightarrow 2Na_2CO_3$ $(aq) + H_2O$ (l)

Chemicals Required:

1. Sodium carbonate $(Na₂CO₃)$ (primary standard)

- 2. Sodium hydroxide solution (unknown concentration)
- 3. Phenolphthalein indicator
- 4. Distilled water

Apparatus Required:

- 1. Burette (50 mL)
- 2. Pipette (25 mL)
- 3. Conical flask (250 mL)
- 4. Volumetric flask (250 mL)
- 5. Beakers (100 mL and 250 mL)
- 6. Clamp stand
- 7. Funnel
- 8. White tile

Procedure

A. Preparation of Standard Sodium Carbonate Solution:

Weigh 2.65 g of anhydrous sodium carbonate accurately using an analytical balance. Transfer the sodium carbonate into a 250 mL volumetric flask with the help of a funnel. Add approximately 100 mL of distilled water to dissolve the sodium carbonate completely by swirling. Add more distilled water up to the calibration mark of the flask. Stopper the flask and shake well for uniform mixing.

B. Titration Process:

Rinse the burette with the sodium hydroxide solution and fill it with the same. Ensure there are no air bubbles in the nozzle. Rinse the pipette with the standard sodium carbonate solution and pipette 25 mL of it into a clean conical flask. Add 2-3 drops of phenolphthalein indicator to the solution in the conical flask. The solution will appear pink. Place the conical flask on a white tile for better visibility of the colour change. Start titration by adding sodium hydroxide solution from the burette into the conical flask, drop by drop, while swirling the flask continuously. As the endpoint approaches (faint pink colour disappearing), add the sodium hydroxide solution dropwise to avoid overshooting the endpoint. Stop adding sodium hydroxide when the pink colour just disappears and remains colourless. This indicates the endpoint of the reaction. Record the burette reading. Repeat the titration at least three times to obtain consistent and accurate readings.

Calculation

Indicator: phenolphthalein

$Na_2CO_3 + 2NaOH \rightarrow 2Na_2CO_3 + H_2O$

 $M1\cdot V1\cdot n1=M2\cdot V2\cdot n2$

Where:

- $M1 =$ Molarity of sodium carbonate
- $V1 = Volume of sodium carbonate solution$
- $n1 =$ Number of moles of sodium carbonate (1)
- M2 = Molarity of sodium hydroxide solution
- V2 = Volume of sodium hydroxide solution
- $n2$ = Number of moles of sodium hydroxide (2)

Rearranging to calculate M2,

M2=M1⋅V1⋅n1V2/n2M₂

 $M2=?$

Result:

The molarity of the sodium hydroxide solution is ---------------------M.

2. Estimation of hydrochloric acid using standard oxalic acid

Aim

To estimate the concentration of hydrochloric acid (HCl) solution using standard oxalic acid $(C_2H_2O_4·2H_2O)$ solution by volumetric analysis.

Principle

This experiment is based on the neutralization reaction between oxalic acid, a weak organic acid, and hydrochloric acid, a strong acid. Oxalic acid acts as a primary standard because it is pure, stable, and easily weighed. In the presence of phenolphthalein indicator, the acid reacts with the base to produce water and a salt. The endpoint is indicated by a color change from colorless to pale pink.

The reaction involved is:

C2H2O4⋅**2H2O+2HCl→CO2+2H2O**

Chemicals Required

- 1. Standard oxalic acid solution
- 2. Hydrochloric acid solution (to be estimated)
- 3. Phenolphthalein indicator

- 1. Burette
- 2. Pipette (25 mL)
- 3. Conical flask (250 mL)
- 4. Beaker
- 5. Funnel
- 6. Distilled water
- 7. White tile

Weigh accurately the required amount of oxalic acid $(C_2H_2O_4·2H_2O)$ and dissolve it in a small quantity of distilled water. Transfer it to a standard flask and dilute to the mark with distilled water to prepare a solution of known molarity (e.g., 0.05 M). Rinse the burette with hydrochloric acid solution and the pipette with the standard oxalic acid solution. Rinse the conical flask with distilled water. Fill the burette with the hydrochloric acid solution and ensure there are no air bubbles in the nozzle, then record the initial burette reading. Using a pipette, transfer 25 mL of the standard oxalic acid solution into the conical flask. Add 2–3 drops of phenolphthalein indicator to the oxalic acid solution in the conical flask. Place the conical flask on a white tile to observe the color change clearly. Slowly add the hydrochloric acid solution from the burette to the oxalic acid solution while swirling the flask gently. Continue adding until a pale pink color persists for 30 seconds, indicating the endpoint. Note the final burette reading. Repeat the titration process until you obtain concordant readings.

Calculation

Indicator: phenolphthalein

C2H2O4⋅**2H2O+2HCl→CO2+2H2O**

$$
M1\cdot V1\cdot n1 = M2\cdot V2\cdot n2
$$

$$
M2 = M1\cdot V1\cdot n1V2/n2M_2
$$

 $M2=?$

Result

The molarity of the hydrochloric acid solution is determined to be ---------------M.

3. Estimation of ferrous sulphate using standard Mohr's salt

Aim

To estimate the concentration of ferrous sulphate (FeSO₄) solution using standard Mohr's salt (ammonium iron (II) sulphate) solution by volumetric analysis.

Principle

This experiment is based on the redox titration between ferrous ions ($Fe²⁺$) in ferrous sulphate and the ferric ions ($Fe³⁺$) present in Mohr's salt, in the presence of potassium dichromate ($K_2Cr_2O_7$) as the titrant. Mohr's salt acts as a primary standard because it is stable, non-hygroscopic, and easily weighed. The reaction involves the oxidation of ferrous ions to ferric ions. The endpoint is detected using a diphenylamine indicator, which changes color from colorless to purple as the iron(III) ions are reduced to iron(II) ions.

The reaction involved is:

$$
6Fe^{2+}+Cr_2O_7^{2-}+14H^+\rightarrow 2Fe_2^{3+}+7H_2O+2Cr^{3+}
$$

Chemicals Required

- 1. Standard Mohr's salt solution
- 2. Ferrous sulphate solution (to be estimated)
- 3. Potassium dichromate $(K_2Cr_2O_7)$ solution
- 4. Diphenylamine indicator
- 5. Dilute sulfuric acid (H_2SO_4)

- 1. Burette
- 2. Pipette (25 mL)
- 3. Conical flask (250 mL)
- 4. Beaker
- 5. Funnel
- 6. Distilled water
- 7. White tile

 Weigh accurately the required amount of Mohr's salt (ammonium iron(II) sulphate) and dissolve it in distilled water to prepare a standard solution of known molarity. Rinse the burette with potassium dichromate solution and the pipette with the standard Mohr's salt solution. Rinse the conical flask with distilled water. Fill the burette with the potassium dichromate solution and ensure there are no air bubbles in the nozzle, then record the initial burette reading. Using a pipette, transfer 25 mL of the ferrous sulphate solution into the conical flask. Add a few drops of diphenylamine indicator to the solution in the conical flask. Add 10 mL of dilute sulfuric acid to the conical flask to ensure an acidic medium. Place the conical flask on a white tile to observe the color change clearly. Slowly add the potassium dichromate solution from the burette to the ferrous sulphate solution while swirling the flask gently. Continue adding the titrant until a purple color persists for 30 seconds, indicating the endpoint. Note the final burette reading. Repeat the titration process until you obtain concordant readings.

Calculation

Indicator: Diphenylamine

6Fe2++Cr2O⁷ 2−+14H⁺→2Fe² 3++7H2O+2Cr3+

M1⋅V1⋅n1=M2⋅V2⋅n2

M2=M1⋅V1⋅n1V2/n2M₂

$M2=?$

Result

The molarity of the ferrous sulphate solution is determined to be ______________ M.

4. Estimation of oxalic acid using standard ferrous sulphate

Aim

To estimate the concentration of oxalic acid solution using standard ferrous sulphate (FeSO₄) solution by volumetric analysis.

Principle

This experiment is based on the redox titration between oxalic acid, a weak organic acid, and ferrous sulphate (Fe^{2+}) , where ferrous ions act as the reducing agent. The ferrous ions are oxidized to ferric ions (Fe^{3+}) in the presence of a suitable oxidizing agent like potassium dichromate $(K_2Cr_2O_7)$. The reaction is conducted in an acidic medium, and the endpoint is indicated by a color change due to the oxidation of iron (II) to iron (III) ions, which is detected using an appropriate indicator, such as diphenylamine.

The reaction involved is:

$$
C_2H_2O_4 + Fe^{2+} \rightarrow Fe^{3+} + CO_2 + H_2O
$$

Chemicals Required

- 1. Standard ferrous sulphate solution
- 2. Oxalic acid solution (to be estimated)
- 3. Potassium dichromate $(K_2Cr_2O_7)$ solution
- 4. Diphenylamine indicator
- 5. Dilute sulfuric acid (H_2SO_4)

- 1. Burette
- 2. Pipette (25 mL)
- 3. Conical flask (250 mL)
- 4. Beaker
- 5. Funnel
- 6. Distilled water
- 7. White tile

Weigh accurately the required amount of ferrous sulphate (FeSO₄) and dissolve it in distilled water to prepare a standard solution of known molarity. Rinse the burette with potassium dichromate solution and the pipette with the standard ferrous sulphate solution. Rinse the conical flask with distilled water. Fill the burette with the potassium dichromate solution and ensure there are no air bubbles in the nozzle, then record the initial burette reading. Using a pipette, transfer 25 mL of the oxalic acid solution into the conical flask. Add a few drops of diphenylamine indicator to the oxalic acid solution in the conical flask. Add 10 mL of dilute sulfuric acid to the conical flask to maintain an acidic medium. Place the conical flask on a white tile to observe the color change clearly. Slowly add the potassium dichromate solution from the burette to the oxalic acid solution while swirling the flask gently. Continue adding the titrant until a pink color persists for 30 seconds, indicating the endpoint. Note the final burette reading. Repeat the titration process until you obtain concordant readings.

Calculation

Indicator: Diphenylamine

$C_2H_2O_4+Fe^{2+} \rightarrow Fe^{3+}+CO_2+H_2O$

 $M1\cdot V1\cdot n1=M2\cdot V2\cdot n2$

M2=M1⋅V1⋅n1V2/n2M₂

$M2=?$

Result

The molarity of the oxalic acid solution is determined to be _______________ M.

5. Estimation of potassium permanganate using standard sodium hydroxide

Aim

To estimate the concentration of potassium permanganate (KMnO4) solution using standard sodium hydroxide (NaOH) solution by volumetric analysis.

Principle

This experiment is based on the redox reaction between potassium permanganate and sodium hydroxide. Potassium permanganate acts as a strong oxidizing agent in alkaline medium, and it reacts with sodium hydroxide to form manganate $(MnO₄²)$. The reaction proceeds until the endpoint, which is indicated by a faint green color of manganate ions in the solution.

The reaction involved is:

2KMnO₄+2NaOH→2KNaMnO₄+H₂O

Chemicals Required

- 1. Standard sodium hydroxide (NaOH) solution
- 2. Potassium permanganate $(KMnO₄)$ solution (to be estimated)
- 3. Distilled water

- 1. Burette
- 2. Pipette (25 mL)
- 3. Conical flask (250 mL)
- 4. Beaker
- 5. Funnel
- 6. White tile

 Weigh accurately the required amount of sodium hydroxide (NaOH) and dissolve it in a small quantity of distilled water. Transfer it to a standard flask and dilute to the mark with distilled water to prepare a solution of known molarity (e.g., 0.05 M). Rinse the burette with potassium permanganate solution and the pipette with the standard sodium hydroxide solution. Rinse the conical flask with distilled water. Fill the burette with the potassium permanganate solution and ensure there are no air bubbles in the nozzle, then record the initial burette reading. Using a pipette, transfer 25 mL of the standard sodium hydroxide solution into the conical flask. Slowly add the potassium permanganate solution from the burette to the sodium hydroxide solution in the conical flask while swirling the flask gently. Continue adding until a faint green color persists for 30 seconds, indicating the endpoint. Note the final burette reading. Repeat the titration process until you obtain concordant readings.

Calculation

Indicator: Self-indicator (KMnO₄ is a colored compound and acts as its own indicator)

2KMnO₄+2NaOH→2KNaMnO₄+H₂O

$M1\cdot V1\cdot n1=M2\cdot V2\cdot n2$

$M2=M1\cdot V1\cdot n1V2/n2M_2$

$M2=?$

Result

The molarity of the potassium permanganate solution is determined to be ____________ M.

6. Estimation of magnesium using EDTA

Aim

To estimate the concentration of magnesium ions (Mg^{2+}) in a given solution using ethylenediaminetetraacetic acid (EDTA) as the titrant by complexometric titration.

Principle

This experiment is based on the formation of a stable, water-soluble complex between magnesium ions $(Mg²⁺)$ and EDTA in an alkaline medium. The reaction proceeds quantitatively at a pH of 10, maintained by an ammonia buffer solution. Eriochrome Black T is used as an indicator, which forms a weak complex with Mg^{2+} , imparting a wine-red color to the solution. As EDTA is added, it replaces the indicator by forming a stronger complex with Mg^{2+} , and at the endpoint, the solution turns from wine-red to sky blue, indicating the completion of the reaction.

The reaction involved is:

Mg2++EDTA4−→[Mg-EDTA]2−

Chemicals Required

- 1. Magnesium ion solution (to be estimated)
- 2. Standard EDTA solution
- 3. Eriochrome Black T indicator
- 4. Ammonia buffer solution (pH 10)
- 5. Distilled water

- 1. Burette
- 2. Pipette (25 mL)
- 3. Conical flask (250 mL)
- 4. Beaker
- 5. Funnel

6. White tile

Procedure

 Prepare a standard EDTA solution by weighing the required amount of disodium EDTA and dissolving it in distilled water to a known volume in a standard flask. Rinse the burette with the EDTA solution and the pipette with the magnesium ion solution. Rinse the conical flask with distilled water. Fill the burette with the EDTA solution and remove air bubbles in the nozzle, then record the initial burette reading. Using a pipette, transfer 25 mL of the magnesium ion solution into the conical flask. Add 10 mL of ammonia buffer solution to maintain the pH at 10. Add 2–3 drops of Eriochrome Black T indicator to the conical flask. The solution will turn wine-red. Titrate the magnesium ion solution with EDTA solution from the burette, swirling the flask gently. Continue adding the EDTA solution until the wine-red color changes to a stable sky-blue color, indicating the endpoint. Note the final burette reading. Repeat the titration process until concordant readings are obtained.

Calculation

S. No.	Volume of magnesium solution (mL)	Burette Readings (mL)		Volume of
		Initial	Final	$EDTA$ (mL)

 Indicator: Eriochrome Black T

Mg2++EDTA4−→[Mg-EDTA]2−

$$
M1\cdot V1\cdot n1 = M2\cdot V2\cdot n2
$$

$$
M2 = M1\cdot V1\cdot n1V2/n2M_2
$$

$M2=?$

Result

The molarity of the magnesium ion solution is determined to be _____________ M.

7. Estimation of ferrous ion using diphenyl amine as indicator

Aim

To estimate the concentration of ferrous ions (Fe^{2+}) in the given solution using potassium dichromate ($K_2Cr_2O_7$) as the titrant and diphenylamine as the indicator.

Principle

The estimation of ferrous ions is based on the redox reaction between potassium dichromate, a strong oxidizing agent, and ferrous ions. Potassium dichromate $(K_2Cr_2O_7)$ in acidic medium oxidizes Fe²⁺ to Fe³⁺, while itself being reduced to Cr³⁺. The reaction occurs quantitatively in an acidic medium provided by dilute sulfuric acid.

Diphenylamine is used as an internal redox indicator, which changes from colorless to a violet color at the endpoint. The indicator works by getting oxidized slightly after the completion of the reaction, indicating the endpoint.

The reaction involved is:

$$
6Fe^{2+}+Cr_2O_7^{2-}+14H^+\rightarrow 6Fe^{3+}+2Cr^{3+}+7H_2O
$$

Chemicals Required

- 1. Potassium dichromate solution (standard solution)
- 2. Ferrous ion solution (to be estimated)
- 3. Diphenylamine indicator
- 4. Dilute sulfuric acid (H_2SO_4)
- 5. Distilled water

- 1. Burette
- 2. Pipette (25 mL)
- 3. Conical flask (250 mL)
- 4. Beaker
- 5. Funnel
- 6. White tile

 Prepare a standard potassium dichromate solution by dissolving the required amount of $K_2Cr_2O_7$ in distilled water and diluting it to the desired volume in a standard flask. Rinse the burette with potassium dichromate solution and the pipette with the ferrous ion solution. Rinse the conical flask with distilled water. Fill the burette with the potassium dichromate solution and ensure no air bubbles are in the nozzle. Record the initial burette reading. Using a pipette, transfer 25 mL of the ferrous ion solution into the conical flask. Add about 10 mL of dilute sulfuric acid to the conical flask to provide an acidic medium. Add 2–3 drops of diphenylamine indicator to the ferrous ion solution in the conical flask. The solution will remain colorless initially. Titrate the ferrous ion solution with potassium dichromate solution from the burette, swirling the flask gently. As the endpoint approaches, the violet color appears momentarily and fades upon mixing. The endpoint is reached when a stable violet color persists, indicating the complete oxidation of Fe^{2+} to Fe^{3+} . Note the final burette reading. Repeat the titration until concordant readings are obtained.

Calculation

Indicator: Diphenylamine

$$
6Fe^{2+}+Cr_2O_7^{2-}+14H^+\rightarrow 6Fe^{3+}+2Cr^{3+}+7H_2O
$$

M1⋅V1⋅n1=M2⋅V2⋅n2 M2=M1⋅V1⋅n1V2/n2M₂

$M2=?$

Result

The molarity of the ferrous ion solution is determined to be _______________ M.