

Microscale Chemistry Laboratory

Science Faculty

Introduction

Microscale chemistry experiments use small quantities of chemicals and simple equipments. These have the advantages of reduced costs, reduced safety hazards and allowing many experiments to be done quickly. For the past ten years, we have been doing Microscale chemistry experiments at our school – using one or two drops of each reagent.

Advantages of Microscale Apparatus

Working at this scale has many advantages:

- Being able to handle small glassware and small quantities of chemicals.
- Having the opportunity to repeat the experiment when the reaction does not work well.
- Better control of organic syntheses when the amount of chemicals is small.
- Saving time and generating less waste.
- Encouraging students to be considerate about environment.
- Leading to a lot of cost reduction.
- Reducing the risks of fire hazards and acid burns to minimum.

Safety and Waste

We do not raise any particular safety precautions to be taken into consideration. Students should wear standard safety equipment, such as laboratory coat, safety glasses, and gloves.

Traditionally, experiments in organic chemistry are carried out on a **macroscale** level, employing quantities of chemicals approximately 5-100 g, using glassware designed to contain between 25 and 500 mL of liquids. For quantities of materials in the 0.005--0.5 gram range, one employs different, “**microscale**” techniques and equipments in order to carry out the various standard organic laboratory operations.

Basic Equipment

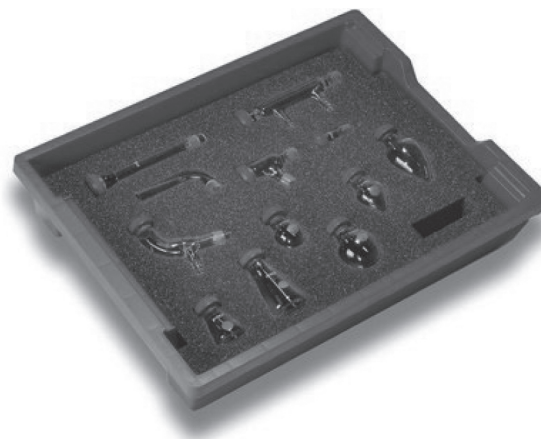
The glassware used for microscale experiments is contained in a Microscale Chemistry Laboratory (MCL) Kit

What is Microscale Chemistry Laboratory (MCL) Kit?

The Microscale Chemistry Laboratory Kit enables students to perform experiments in an environmentally safe and pollution-free atmosphere, using small quantities of chemicals without compromising with the quality and standard of experiments. The experiments can be performed easily and quickly. Some of the contents of the microscale kit are:

Science Faculty, Sadhu Vaswani International School for Girls, New Delhi.

1. Micro burette – 10 mL
2. Micro measuring cylinder – 10 mL
3. Micro beaker – 10 mL
4. Micro test tube – 2mL
5. Micro test tube wooden handle
6. Petri dish – 10 cm in diameter
7. Micro funnel
8. Micro conical flask – 25 mL



Aim

To determine the strength of KMnO_4 solution by titrating it against a standard solution of Ferrous Ammonium Sulphate (Mohr's salt) by Microscale Chemistry Laboratory Techniques as well as Conventional Chemistry Laboratory Techniques

Theory

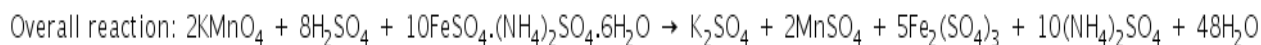
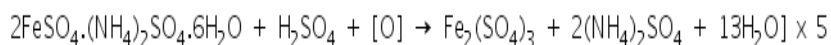
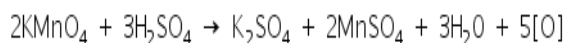
In redox systems, the titration method can be adopted to determine the strength of a reductant/oxidant using a redox sensitive indicator. Redox titrations, involving potassium permanganate, are called permanganometric titrations. In these reactions, MnO_4^- ions act as the self indicator.

In this titration, potassium permanganate is the oxidizing agent and Mohr's salt is the reducing agent. Mohr's salt is a double salt of Ferrous Sulphate and Ammonium Sulphate and its composition is $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$.

In this titration, the MnO_4^- ion is reduced to Mn^{2+} in the presence of acid and Fe^{2+} ions of Mohr's salt are oxidized to Fe^{3+}

The chemical reaction that occurs in this titration can be represented by the following chemical equations.

Molecular Equation



Balanced Chemical Equation

From the overall balanced chemical equation, it is clear, that 2 moles of potassium permanganate react with 10 moles of Mohr's salt.

$$\text{Therefore, Molarity of KMnO}_4 = \frac{\text{Molarity of Mohr's salt} \times \text{Volume of Mohr's salt} \times 2}{\text{Volume of KMnO}_4 \times 10}$$

Conventional Method (For a batch of 20 students)

Apparatus Required: 50 mL burette, 250 mL volumetric flask, 20 mL pipette, funnel, weighing bottle, clamp stand, titration flask.

Chemicals Used: Potassium Permanganate solution (KMnO₄), Ferrous Ammonium Sulphate (Mohr's salt), diluted H₂SO₄ and Conc. H₂SO₄

A. To Prepare M/100 KMnO₄ Solution

Molecular weight of KMnO₄ = 158 g

In 1000 mL, we dissolve 1.58 g of KMnO₄ to prepare M/100 solution for titration.

Each student can use up to 50 mL of KMnO₄ for 3 to 4 titrations.

Volume to be used in Burette = 50 mL

B. To Prepare 250 ml of M/20 Mohr's Salt Solution

Volumetric flask used = 250 mL

1000 mL requires 392 g of Mohr's salt to prepare 1 M solution.

$$\begin{aligned} \text{For 250 mL, Mohr's salt required} &= \frac{392 \times 250}{1000} \\ \text{To prepare 100 mL of M/20 solution, we need} &= \frac{392 \times 250}{1000 \times 20} \\ &= 4.9 \text{ g} \end{aligned}$$

Each student will weigh 4.9 g to make 250 mL of Mohr's salt solution.

20 students will use $20 \times 4.9 = 98 \text{ g}$

10 mL of Sulphuric Acid added before titration to Mohr's salt solution.

Observation Table

S. No	Volume of Mohr's salt used (mL)	Burette reading/ml		Volume of KMnO_4 solution used ($V_2 - V_1$)/mL
		Initial (V_1)	Final (V_2)	
1.	20	0.0	21.1	21.1
2.	20	21.1	42.2	21.1
3.	20	0.0	21.1	21.1

Calculation:

Volume of Mohr's salt = 20 mL

Molarity of Mohr's salt = M/20

Volume of KMnO_4 solution = 21.1 mL

Molarity Equation

$$\frac{(\text{Volume of } \text{KMnO}_4 \times \text{Molarity of } \text{KMnO}_4)}{(\text{Volume of Mohr's salt} \times \text{Molarity of Mohr's salt})} = \frac{(\text{Number of moles of } \text{KMnO}_4)}{(\text{Number of moles of Mohr's salt})}$$

$$= \frac{21.1 \times \text{Molarity of } \text{KMnO}_4}{20 \times 0.05}$$

$$= \frac{2}{10}$$

Therefore, molarity of the given KMnO_4

$$\frac{20 \times 0.05 \times 2}{21.1 \times 10}$$

$$= 0.0095\text{M}$$

Molar mass of KMnO_4 = 158g/mol

Therefore,

The strength of the given KMnO_4 = Molarity of KMnO_4 $\times$ Molar mass of KMnO_4

$$= 0.0095 \times 158$$

$$= 1.501 \text{ g/ litre}$$

Result: The strength of the given KMnO_4 solution = 1.501 g/ litre

Using Microscale Apparatus

Apparatus: Two 5 mL burettes, 50 mL volumetric flask, funnel, weighing bottle, clamp stand, titration flask.

Chemicals Required: Potassium Permanganate solution (KMnO_4), Ferrous Ammonium Sulphate (Mohr's salt), diluted H_2SO_4 and Conc. H_2SO_4

A. To Prepare M/100 KMnO_4 Solution

Molecular weight of $\text{KMnO}_4 = 158 \text{ g}$

In 1000 mL, we dissolve 1.58 g of KMnO_4 to prepare M/100 solution for titration.

To prepare 250 mL of M/100 KMnO_4 , we need 0.395 g of KMnO_4

Each student can use up to 10 mL of KMnO_4 for 3 to 4 titrations.

Volume to be used in Burette = 5 mL

B. To Prepare 50 ML OF M/20 Mohr's Salt Solution

1000 ml requires 392 g of Mohr's salt to prepare 1 M solution.

For 50 mL, Mohr's salt required = $\frac{392 \times 50}{1000}$

To prepare 50 ml of M/20 solution, we need = $\frac{392 \times 50}{1000 \times 20}$
= 0.98 g

Each student will weigh 0.98 g to make 50 ml of Mohr's salt solution.

20 students will use $20 \times 0.98 = 19.6 \text{ g}$

2 ml of Sulphuric Acid added to solution of Mohr's Salt before titration.

Observation Table

S. No	Volume of Mohr's salt solution used/ mL	Burette reading/ml		Volume of KMnO_4 solution used (V2- V1)/ mL
		Initial (V1)	Final (V2)	
1.	2	0.0	2.1	2.1
2.	2	2.1	4.2	2.1
3.	2	4.2	6.3	2.1

Calculation:

Volume of Mohr's salt = 2 mL

Molarity of Mohr's salt = M/20

Volume of KMnO_4 solution = 2.0 mL

Molarity Equation

$$\frac{(\text{Volume of KMnO}_4 \times \text{Molarity of KMnO}_4)}{(\text{Volume of Mohr's salt} \times \text{Molarity of Mohr's salt})} = \frac{(\text{Number of moles of KMnO}_4)}{(\text{Number of moles of Mohr's salt})}$$

$$= \frac{2.0 \times \text{Molarity of KMnO}_4}{2 \times 0.05}$$

$$= \frac{2}{10}$$

Therefore, molarity of the given KMnO_4 $\frac{20 \times 0.05 \times 2}{21.1 \times 10}$

$$= 0.0095\text{M}$$

Molar mass of $\text{KMnO}_4 = 158 \text{ g/mol}$

Therefore,

The strength of the given $\text{KMnO}_4 = \text{Molarity of KMnO}_4 \times \text{Molar mass of KMnO}_4$

$$= 0.0095 \times 158$$

$$= 1.501 \text{ g/ litre}$$

Result: The strength of the given KMnO_4 solution = 1.501 g/ litre

Conclusion

- From the Results, we can conclude that the strength of unknown KMnO_4 solution was determined by few grams of solid and few drops of solution in microscale chemistry laboratory compared to the conventional chemistry laboratory. Therefore, the amount of chemical used in microscale laboratory is minimal.
- A typical microscale experiment will consume only 1% of the required quantity of reagents used in a traditional macro scale experiment.
- There is lot of cost reduction and breakage is minimised in microscale chemistry laboratory
- The developed experiments are easy to implement and require equipment often available in organic chemistry labs with very low consumption of chemicals. By using small amounts of chemicals, the reactions proceed quickly and generate less waste, which is beneficial both financially and environmentally.

By adopting this teaching strategy, the laboratory becomes less consumer of chemicals, cleaner, more secure and less polluting while ensuring students' better adherence to the practical learning process.