Preparation Of 250 ml Of N/20 Solution Of Mohr's Salt

Theory

The molecular formula of Mohr's salt is $FeSO_4.(NH_4)_2SO_4.6H_2O$.

The ionic equation for the oxidation of Mohr's salt is

$$Fe^{2+} \longrightarrow Fe^{3+} + e^-$$

It is clear from the above equation that one electron is given out during the oxidation of one molecule of Mohr's salt.

$$\therefore \text{ Equivalent mass of Mohr's salt} = \frac{\text{Molecular mass of Mohr's salt}}{\text{Number of electrons lost by one molecule of it}}$$

$$= \frac{392}{1} = 392$$

$$\text{Strength (g/litre)} = \text{Normality} \times \text{Eq. mass}$$

$$= \frac{1}{20} \times 392 = 19.6 \text{ g/litre}$$

For preparing 250 ml of $\frac{N}{20}$ Mohr's salt solution Mohr's salt needed

$$= \frac{19.6}{1000} \times 250 = 4.9 \text{ g}$$

Apparatus

Watch glass, weight box, fractional weight box, 250 ml beaker, glass rod, 250 ml measuring flask and wash bottle.

Chemical Required

Mohr's salt, cone. H_2SO_4 and distilled water.

Procedure

1. Weigh the clean and dry watch glass and record its weight in the note-book.
2. Weigh accurately 4.9 g of Mohr's salt crystals on the watch glass and record the weight in the note-book.
3. Transfer carefully the weighed Mohr's salt from the watch glass into a clean 250 ml beaker. Add to this beaker about 5 ml of cone, sulphuric acid to check the hydrolysis of ferrous sulphate.
4. Wash the watch glass thoroughly with distilled water to transfer the sticking salt completely into the beaker. Dissolve the salt in the beaker with gentle stirring.
5. Transfer the entire solution carefully into the 250 ml measuring flask through a funnel.

6. Wash the beaker with distilled water and transfer the washing's into the measuring flask.
7. Add enough distilled water to the measuring flask carefully up to just below the etched mark on its neck with the help of wash bottle.
8. Add the last few drops of distilled water with a pipette until the lower level of the meniscus just touches the mark on the measuring flask.
9. Stopper the measuring flask and shake it gently to make the solution homogeneous (i.e., uniform throughout) and label it as M/20 Mohr's salt solution.

Law of Equivalents

According to this law, the number of equivalents of the substance to be titrated (litre) is equal to the number of equivalents of the titrant used. Derivation of the normality equation. Consider an acid alkali neutralization reaction. Let Vx cm3 of aq add solution of normality requires V2 cm3 of base of N2 normality for complete neutralization.

We know that 1000 cm^3 of 1N acid solution contains acid = 1 gram equivalent.

V_1 cm^3 of N_1 acid contains acid = $\frac{1}{1000} \times V_1 \times N_1$ gm equivalents. Thus, number of gram equivalents of acid in V_1 cm^3 solution = $\frac{V_1N_1}{1000}$.

Similarly, number of gram equivalents of base in V_2 cm^3 of its N_2 solution = $\frac{V_2N_2}{1000}$.

By the law of equivalents, at the end point, $\frac{V_1N_1}{1000} = \frac{V_2N_2}{1000}$.

$$\boxed{N_1V_1 = N_2V_2}$$

It is known as **normality equation.** If three factors (V_1, V_2, N_1) are known, the fourth (N_2) can be calculated by using above formula.

In terms of molarities we can proceed as

$$\frac{[\text{Molarity } (M_1) \times \text{Volume } (V_1)] \text{ of Acid}}{[\text{Molarity } (M_2) \times \text{Volume } (V_2)] \text{ of Base}}$$

$$= \frac{\text{Numerical coefficient of acid in the balanced equation}}{\text{Numerical coefficient of base in the balanced equation}}$$

For a reaction between HCl and Na_2CO_3

$$Na_2CO_3 + 2HCl \longrightarrow 2NaCl + CO_2 + H_2O$$

Thus, $$\frac{(\text{Molarity} \times \text{Volume}) \text{ of HCl}}{(\text{Molarity} \times \text{Volume}) \text{ of } Na_2CO_3} = \frac{2}{1}$$

The normality relation and the molarity relation are not restricted to acid-base titrations but are applicable to all types of reactions.

Process of Titration

The process of titration is employed to find out the volume of one solution required to react completely with a certain known volume of solution of some other substance. This is the most important step in volumetric analysis. The process of titration is carried out as under:

1. Support a cleaned and rinsed burette with a burette clamp. Close the stopcock and, with the help of a funnel, fill the burette to just above the zero mark. Open the stop cock briefly to remove any air bubbles in the tip.
2. Place a glazed white tile below the burette and place the titration flask on the glazed tile below the burette nozzle. Adjust the height of the burette so that the nozzle tip just enters the mouth of the titration flask.
3. Note initial reading of the burette and run out the solution from the burette (one ml at a time}. During titration, operate the stopcock with your left hand and constantly swirl the flask with the right hand. (See Fig).

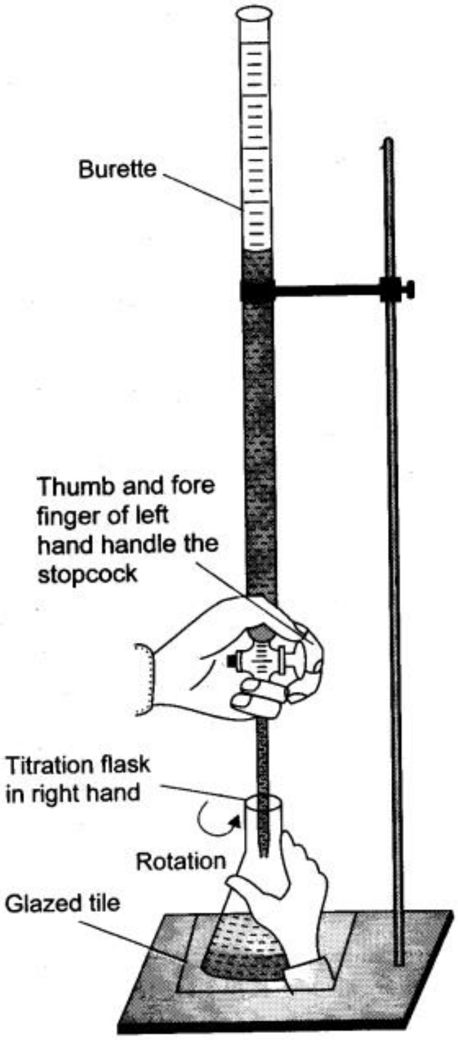
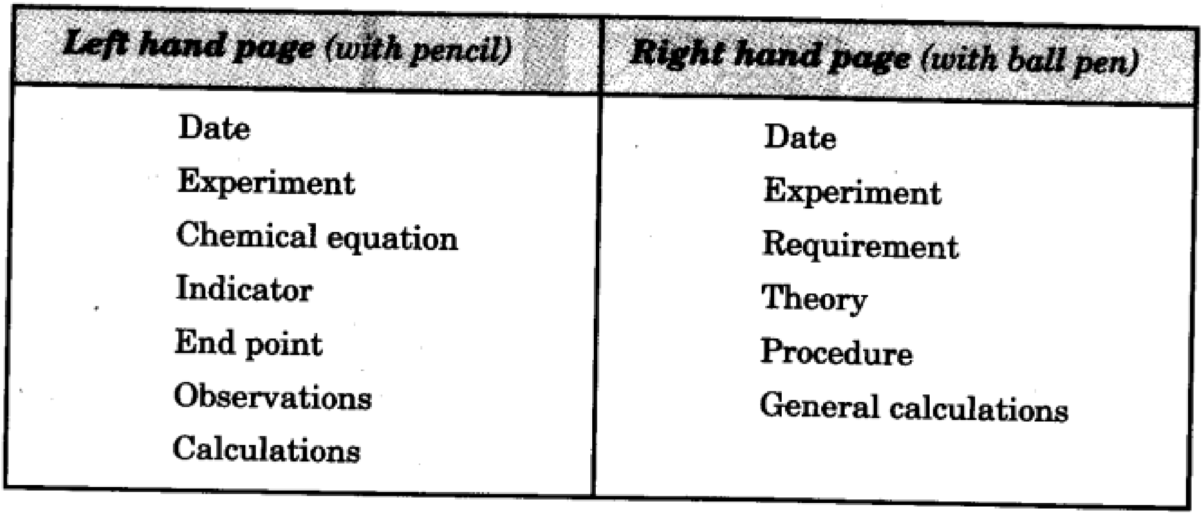


Fig. Correct way of handling a burette.

4. Continue running more of the solution from the burette into the titration flask. The solution should fall directly into the solution of titration flask. It should not fall on the walls of flask.
5. Stop addition of the solution when the end point is reached and take final reading of the burette. The difference between the final and initial readings gives rough volume of the solution used for completion of the reaction.
6. The solution from the titration flask is thrown in the sink and the titration flask is washed thoroughly first by keeping it under tap water and then with a little of distilled water. Do not rinse the titration flash.
7. Pour more solution in the burette.
8. Pipette out 20 ml of the solution into the titration flask and add a drop or two of the indicator solution.
9. Take initial reading of the burette. Run solution from the burette into the titration flask slowly with constant shaking. Continue running of the solution till the volume added is 1 ml less than the rough volume found out in the first titration. Now add solution from the burette drop wise. (Add a drop, close the pinch cock, shake and find out if the end point has been attained).
10. Continue adding solution drop wise from the burette, till by addition of last single drop, the end point is attained.
11. Note down the final reading of the burette. The difference between the final and initial readings of the burette gives the exact volume of the solution required for completion of the reaction.
12. Check the correctness of the end point by adding one drop of solution (taken in the titration flask) with the help of a pipette. Restoration of original colour confirms the correctness of the end point.
13. Perform 5-6 titrations so that at least three concordant readings (difference not more than 0.05 ml) are obtained.

Recording of Volumetric Analysis in the Practical Note Book



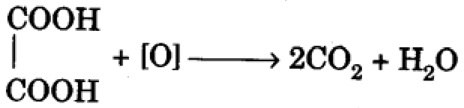
Left hand page *(with pencil)*	***Right hand page*** *(with ball pen)*
Date	**Date**
Experiment	**Experiment**
Chemical equation	**Requirement**
Indicator	**Theory**
End point	**Procedure**
Observations	**General calculations**
Calculations	

Experiments on Potassium Permanganate Titrations -Permanganometric Titrations

Potassium permanganate is a strong oxidizing agent in the presence of sulphuric acid

$$2KMnO_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$$

Potassium permanganate can be used for the estimation of oxalic acid (or oxalates) and ferrous salts. It oxidizes oxalic acid to CO_2 and ferrous salts to ferric salts in acidic medium



$$\begin{array}{l} COOH \\ | \\ COOH \end{array} + [O] \longrightarrow 2CO_2 + H_2O$$

$$2FeSO_4 + H_2SO_4 + [O] \longrightarrow Fe_2(SO_4)_3 + H_2O$$

In $KMnO_4$ titrations, no external indicator is required. ***$KMnO_4$ acts as self indicator.*** The end point is appearance of permanent pinkish tinge with the last single drop of $KMnO_4$ solution.

Important Instructions for KMn04 Titrations

1. $KMnO_4$ solution is always taken in the burette.
2. Avoid the use of a burette having a rubber tap as $KMnO_4$ attacks rubber.
3. Add about an equal volume of dil. H_2SO_4 (- 4N) to the solution to be titrated (say a full test tube for 20 ml of the solution) before adding $KMnO_4$. HCl cannot be used as it gets oxidized to Cl_2 by $KMnO_4$. HNO_3 also cannot be used as it itself is a strong oxidizing agent.
4. If oxalic acid or some oxalate is to be titrated, add required amount of dil. H_2SO_4 and heat the flask to 60°—70°C on a wire gauge. In order to get some idea about the temperature of the solution touch the flask to the back side of your hand (Fig). When it becomes just unbearable to touch, the required temperature is reached. The purpose of heating is to increase the rate of reaction which otherwise is slow at room temperature.

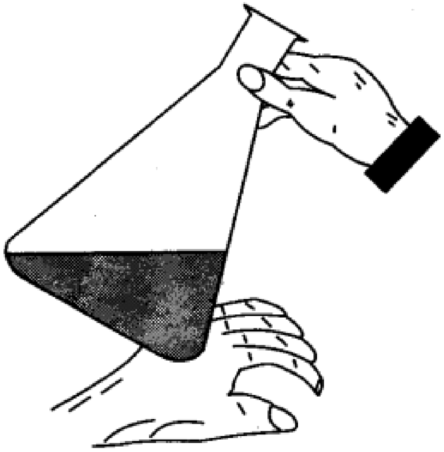


Fig. How to observe approximate temperature of the solution.

5. In case of ferrous salts, no warming is required.
6. Read the upper meniscus while taking burette reading with $KMnO_4$ solution.
7. In case, on addition of $KMnO_4$ a brown ppt. appears, this shows that either H_2SO_4 has not been added or has been added in insufficient amount. In such a case, throw away the solution and titrate again.
8. Potassium permanganate does not dissolve into water readily. It is dissolved by the process of extraction. Transfer the weighed $KMnO_4$ into a beaker and add into it 20-30 ml of distilled water and stir. Transfer the solution into a measuring flask. Add more distilled water (20-30 ml) into the beaker and repeat the operation till the permanganate completely dissolves. Add more distilled water into the measuring flask till the lower meniscus of the solution is in line with the mark on the neck. Stopper the measuring flask and shake to get the solution of uniform strength.