(21)

Experiment No. 4

Aim

To determine the strength of sodium nitrite ($NaNO_2$) in the given water sample provided N/10 $KMnO_4$ solution and N/10 Mohr's salt solution.

Chemical required

N/10 $KMnO_4$ solution, N/10 Mohr's salt solution and 0.75 H_2SO_4

Apparatus required

Burette, pipette, funnel, 250mL flask, burette holder, measuring Cylinder

Indicator

$KMnO_4$ acts as a self-indicator.

End-point

Disappearance of pink colour

Theory

Nitrites can be naturally or artificially occurring in groundwater. Nitrites come from fertilizers through run-off water, sewage and mineral deposits. High levels of nitrites are toxic to humans and animals, especially infants. It can enter the body as nitrate, a nutrient which is essential to plant growth and be converted into nitrite, which disrupts the oxygen delivering ability of hemoglobin in the bloodstream. Infants can develop a life-threatening blood disorder known as blue baby syndrome if exposed to the nitrite contaminated water. Therefore it is important to determine the strength of nitrite ion in the water sample.

The strength of $NaNO_2$ in a given water sample can be measured by titrating the given nitrite solution against standard $KMnO_4$ solution in acidic medium. Acidified $KMnO_4$ oxidizes nitrite ion to nitrate ion and it reduces to manganese ions as per the following reactions:

$$5\,NO_2^- + 5\,H_2O \longrightarrow 5NO_3^- + 10\,H^+ + 10\,e^-$$
$$2\,MnO_4^- + 16H^+ + 10\,e^- \longrightarrow 2Mn^{2+} + 8\,H_2O$$

$$2MnO_4^- + 5\overset{+3}{N}O_2^- + 6H^+ \longrightarrow 2Mn^{2+} + 5\overset{+5}{N}O_3^- + 3H_2O$$

The following problems were observed

1. Nitrite ion is unstable in acidic medium and readily gets converted into nitrous acid which is volatile.

$$NO_2^- + H^+ \longrightarrow HNO_2$$

$$2HNO_2 \longrightarrow NO\uparrow + NO_2\uparrow + H_2O$$

2. Environmental oxidation of NO_2^- may take place and produce an error in the result.
3. The reaction between $KMmO_4$ and NO_2^- is not instantaneous; hence accurate reading cannot be obtained.

In order to avoid these problems, we may change our approach from direct to indirect titration in which a known excess volume of $KMnO_4$ is taken in a conical flask in acidic medium and temperature is raised up to approximately 40^0 C following by addition of a known volume of nitrite solution. At this temperature, the reaction between $KMnO_4$ and NO_2^- is fast and instantaneous as soon as we add the NO_2^- in the conical flask.

The volume of $KMnO_4$ is taken in slight excess and un-reacted $KMnO_4$ is determined by titrating the reaction mixture against N/10 Mohr's salt solution. Acidified $KMnO_4$ solution being oxidizing agent oxidizes Fe^{2+} to Fe^{3+} and itself reduces to Mn^{2+} ions. The redox reaction between Mohr's salt and KMnO4 is as follows:

$$Fe^{2+} \longrightarrow Fe^{+3} + e^- \quad]x5$$

$$MnO_4^- + 8H^+ + 5e^- \longrightarrow Mn_2^+ + 4H_2O$$

$$5Fe_2^+ + MnO_4 + 8H^+ \longrightarrow 5Fe^{3+} + Mn^{2+} + 4\,H_2O$$

In order to overcome the problem for the formation of volatile nitrous acid and environmental oxidation of $NaNO_2$ precaution should be taken that the tip of the pipette should be dipped in the solution while slowly adding nitrite solution, so that no atmospheric oxidation of $NaNO_2$ takes place and NO_2^- ions do not get any time or any chance to change to nitrous acid. In this way, we can determine the amount of $KMnO_4$ left unreacted and hence the amount of $KMnO_4$ used by NO_2^- ions.

Procedure

- Pipette out 10 mL of N/10 $KMnO_4$ and transfer it to 250 mL conical flask
- Add 100 mL of 0.75 N H_2SO_4 with the help of measuring cylinder.
- Warm the resulting solution up to 40^0C (approx).
- Now slowly add 10 mL of nitrite solution to the solution in the conical flask by keeping the tip of the pipette below the surface of the liquid.
- Titrate the resultant solution against N/10 Mohr's salt solution till the disappearance of pink colour.
- Note the volume of Mohr's salt solution consumed
- Repeat the experiment to get concordant reading.
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Observations

S. No.	Initial burette reading	Final burette reading	Vol. of Mohr's salt sol. Used (mL)
1			
2			
3			

Concordant reading = A

Calculation:

Volume of N/10 $KMnO_4$ solution taken each time = 10 mL

Volume of nitrite solution taken each time = 10 mL

Volume of Mohr's salt solution consumed for the unreacted N/10 $KMnO_4$ solution reacting nitrite solution = (10 – A) mL

Therefore, Volume of N/10 $KMnO_4$ = (10 – A) mL

Now, by applying normality equation

$$N_1V_1\ (NaNO_2) = N_2V_2\ (KMnO_4)$$

$$N_1 \times 10 = 1/10 \times V_2$$

$$N_1 \times 10 = 1/10 \times (10 - A)$$

$$N1 = 10\text{-}\ A/100$$

Strength = N1 x Eq. Wt. of $NaNO_2$ x 1000 mg/L

Result:

The strength of $NaNO_2$ solution = ... mg/L or ppm.