Experiment 9
Determination of Total Iron in Water



Introduction:

Iron, as well as manganese, creates serious problems in public water supplies. The problems are most critical for groundwater. Iron exists in soils and minerals as insoluble ferric oxide/hydroxide and iron sulphide (pyrite). In some areas, it also occurs as ferrous carbonate, which is very slightly soluble. Since groundwater usually contains significant amounts of carbon dioxide, appreciable amounts of ferrous carbonate may be dissolved according to Eq.9.1.

$$FeCO_3 + CO_2 + H_2O = Fe^{2+} + 2HCO_3^- \qquad \textbf{9.1}$$

If reducing (anaerobic) conditions exist in groundwater environment, the insoluble ferric iron [Fe^3+] is reduced to more soluble ferrous iron [Fe^2+] and iron concentration in water increases. There seems to be enough evidence to suggest that development of reducing (anaerobic) condition is essential for appreciable amount of iron (as well as manganese) to gain entrance into water.

Environmental significance:

As far as is known, human suffer no harmful effects from drinking waters containing iron and manganese. Such waters, when exposed to the air so that oxygen can enter, become turbid and highly unacceptable from the aesthetic' viewpoint owing to the oxidation of iron and manganese to the Fe^3+, and Mn4+ states which form colloidal precipitates. The rates of oxidation are not rapid, and thus reduced forms can persist for some time in aerated waters. This is especially true when the pH is below 6 with iron oxidation and below 9 with manganese oxidation. The rates may be increased by the presence of certain inorganic catalysts through the action of microorganisms.

Both iron and manganese interfere with laundering operations, impart objectionable stains to plumbing fixtures and cause difficulties in distribution systems by supporting growths of iron bacteria. Iron also imparts a taste to water, which is detectable at very low concentrations.

Guideline:

According to Bangladesh Environment Conservation Rules (2023), drinking water standard for iron is 0.3 - 1.0 mg/l.

Theory on Experimental Method:

Iron may be present in two forms, namely the reduced form (ferrous, Fe^2+) and the fully oxidized form (ferric, Fe^3+). Ferric iron is seldom found in true solution in natural waters, unless they are highly acidic, because of the formation of insoluble ferric hydroxides. Ferrous iron is more likely to be found in true solution, although it is easily oxidized to the ferric state and precipitated in alkaline waters as ferric hydroxide.

Since some iron may exist as iron hydroxide precipitates, therefore it is necessary to bring precipitated form(s) of iron back in 'to solution before oxidizing total iron content in water. For this hydrochloric acid is added to the test sample to dissolve the insoluble ferric forms.

For determination of total iron by the following procedure, it must be ensured that all iron exists in ferric form (Fe^3+). This is most readily accomplished by using potassium permanganate, an oxidizing agent.

$$5Fe^{2+} + MnO_4^- + 8H^+ = 5\,Fe^{3+} + Mn^{2+} + 4H_2O \qquad \textbf{9.2}$$

Ferric iron is determined by producing a red-colored iron compound, ferric thiocyanate, by the addition of potassium thiocyanate (Eq.-9.3).

$$Fe^{3+} + 3\,KCNS = Fe(CNS)_3 + 3\,K^+ \qquad \textbf{9.3}$$
(red)

The quantity of ferric iron is determined by comparison with the red color produced by standard iron solutions.

Apparatus:

1. Nessler tube
2. Measuring cylinder
3. Dropper

Reagents:

1. Hydrochloric acid
2. Potassium permanganate solution
3. Potassium thiocyanate solution
4. Standard iron solution

Procedure:

1. Place 100 mL of the water sample in a Nessler.
2. Add 5 mL of dilute hydrochloric acid
3. Add two drops of potassium permanganate solution
4. Add 5 mL of potassium thiocyanate solution. The solution would turn brown if iron is present.
5. Compare the brown color formed with the standard prepared as follows:
   a. Add 100 mL of distilled water in a Nessler tube
   b. Add 5 mL of the dilute hydrochloric acid.
   c. Add two drops of potassium permanganate solution.
   d. Add 5 mL of potassium thiocyanate solution.
   e. Add 0.2 mL at a time of the standard iron solution until the color of the standard and the sample match.

Calculation:

Total iron concentration of test sample (mg/L) =

$$\frac{\text{Standard iron solution added in distilled water(mL)} \times \text{Conc. of standard iron solution(mg/L)}}{\text{mL of sample taken}}$$

DATA SHEET

Experiment Name :
Experiment Date :

Student's Name :
Student's ID :
Year/ Semester :
Section :
Group :

Table

Sample No	Source of Sample	Temperature of Sample (°C)	Total iron concentration (mg/L)

Signature of the course teacher and date