Course Number : CE 332
Course Name : Environmental Engineering Lab
Course Credit : 1.50

Experiment No. 9
Determination of Iron Concentration of 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 sulfide (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. 1.

$$FeCO_3 + CO_2 + H_2O = Fe^{2+} + 2HCO_3^- \quad (1)$$

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, humans 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 Mn^{4+} states that 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 of through the action of micro-organisms.

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. According to Bangladesh Environment Preservation Act (1997), drinking water standard for iron is 0.3-1.0 mg/l.

THEORY

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.

Ferric iron is determined by producing a red-colored iron compound, ferric thiocyanate, by the addition of potassium thiocyanate (Eq. 2).

$$Fe^{3+} + 3\ KCNS = Fe(CNS)_3 + 3\ K^+ \qquad (2)$$

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

Since some iron may exist as iron hydroxide precipitates, it is necessary to make sure that all iron is in soluble condition. This is done by treating the sample to be tested with hydrochloric acid to dissolve the ferric hydroxide. For determination of total iron following this procedure, it must be ensured that all iron exist in ferric form (Fe^{3+}). This is most readily accomplished by using potassium permanganate, an oxidizing agent.

Ferrous iron is also determined colorimetrically by producing a blue compound, known as Turnbull's blue, by the addition of potassium ferriocyanate (Eq. 3).

$$3\ Fe^{2+} + 2\ K_3Fe(CN)_6 = Fe_3\ [Fe(CN)_6]_2 + 6\ K^+ \qquad (3)$$

The quantity of ferrous is determined by comparison with the color produced by standard iron solutions. Standard iron solutions (in both cases) are prepared from ferrous ammonium sulfate crystals [$FeSO_4(NH_4)_2SO_4.6H_2O$].

REAGENTS

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

APPARATUS

(1) Nessler tube (2)
(2) Measuring cylinder (1)
(3) Dropper (1)

PROCEDURE

(1) Place 100 ml. of the water sample in a Nessler tube.
(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

Iron present (mg/l) = ml. of the standard iron solution used.

ASSIGNMENT QUESTIONS

(1) Discuss the environmental significance of "iron" in water.

(2) Why hydrochloric acid and potassium permanganate are added to the sample in the determination of iron.

(3) Groundwater with high dissolved iron concentration often becomes turbid after extraction (in contact with air). Explain why.