Determination of Particulate Iron

Introduction:

Iron is needful by most living organisms in larger quantities than any other element apart from the structural atoms. Iron is an essential constituent of many enzymatic and other cellular processes. Oxidative metabolism in all organisms and photosynthesis in plants involves cytochromes which contain iron. Iron is needed in the haemoglobin of blood and has a vital role in the nitrogenase enzyme of some lower plants and bacteria.

Organic and inorganic forms of iron exist in particulate and dissolved phases in most waters. The particulates are represented by iron contained in organic matter and hydroxides. In oxygen deficient waters, the reduced form iron (ferrous), which while not toxic, tends to react with air to form floc of hydrated ferric oxide, which can smother developing fish eggs and larvae. Comparing with other trace metals, iron is a les significant contaminant in the environment.

Principle:

The sample is filtered through a standard filter (0.45 µm) that retains particles exceeding its diameter and the filter is treated with hot dilute hydrochloric acid for a few minutes. The iron thus brought into solution is reacted with α,α^1 – dipyridyl in an acetate buffer, in the presence of hydroxylamine, to give an orange ferrous complex and its extinction is measured at 522 nm.

Reagents:

1. Hydroxylamine Hydrochloride: Dissolve 10 gms in 100 ml D.W.

2. Sodium Acetate Buffer: Dissolve 75 g of sodium acetate trihydrate in 100 ml D.W.

3. α,α^1 – Dipyridyl Reagent: Dissolve 0.40 g of α,α^1 – dipyridyl in 2.0 ml of conc. HCl and a little water. Dilute to 100 ml with D.W.

4. Iron Extraction Reagent: 20 ml of conc. HCl diluted to 500 ml.

Experimental Procedure:

Having filtered a suitable volume of sample by using standard filter, place it immediately into a 50 ml stoppered measuring cylinder containing 10±0.5 ml of iron extraction reagent. Filter should be immersed in solution. Place the cylinder on boiling water bath for 10-15 minutes and

allow it to cool at room temperature. Add 1 ml of hydroxylamine hydrochloride solution, followed by 2 ml of sodium acetate buffer and mix the contents. Then, add 1 ml of dipyridyl reagent. Make the volume exactly 50 ml with distilled water. Allow the colour to develop for at least 20 min and read the extinction at 522 nm.

Calibration:

1. Standard Iron Solution:

Dissolve 0.392 g of analytical reagent (AR) quality Ferrous Ammonium Sulphate (FAS) in a little water. Add 2 ml of Conc. HCl and dilute to 100 ml in a volumetric flask.

1 ml = 10 µg-at Fe

For use, dilute 5 ml of above solution to 500 ml with D.W. and do not keep for longer than 1 or 2 days.

1 ml = 0.1 µg-at Fe

2. Procedure:

Add 10 ml of iron extraction reagent to each of six 50 ml stoppered measuring cylinders. Reserve two as blanks and to each of the remaining four, add 5 ml of dilute standard iron solution and carry out the determination as described above.

To calculate F,

$$F = \frac{500}{E_s - E_b}$$

Where, E_s = Mean extinction of four standards

E_b = Mean extinction of two blanks

Calculation: Particulate iron µg-at Fe/L $= \frac{E \times F}{V}$

Where, E = Sample extinction
F = Factor value (5102.04)
V = Volume of sample filtered
Atomic weight of Fe = 56 (55.85)

Result: