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An Improved Field Test for Rapid Estimation of Total Iron in Drinking Water

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The present work discusses the use of bathophenanthroline reagent (in thioglycolic acid) for estimation of total iron. Instant development of light pink to reddish pink colour indicates presence of iron above 0.3 mg/L. The intensity of colour is proportional to the concentration of iron. However, exact concentration of iron can be determined from the colour chart provided therein. Naturally occurring ions present in ground waters do not interfere. The proposed test is quantitative, rapid, sensitive and easy, hence it can be incorporated in the existing water testing field kits.

Key Words: Total iron, Bathophenanthroline reagent, Colour chart.

INTRODUCTION

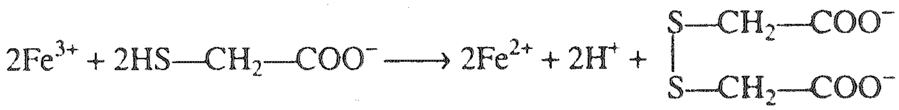
Although iron has got little concern as a health hazard but is still considered as a nuisance in excessive quantities[1–4]. Iron in excess of 0.3 mg/L causes staining of clothes and utensils. The higher concentration of iron is also not suitable for processing of food, beverages, ice, dyeing, bleaching and many other items. Water with higher concentration of iron if used in preparation of tea and coffee, interacts with tannins to give a black inky appearance with a metallic taste. Coffee may even become unpalatable at concentration of iron more than 1 mg/L. Potatoes also turn black on boiling in such types of water. Iron in higher concentration may also cause vomitting. The limits on iron in water are based on aesthetic and taste consideration rather than its physiological effects. The permissible limit[5, 6] for iron(II) in drinking water is 0.3 mg/L.

Iron occurs in two oxidation states: iron(II) (ferrous) and iron(III) (ferric). Reduced iron is generally more soluble than oxidized iron. In ground waters, most of the iron remains in ferrous state due to general lack of oxygen. In alkaline conditions in groundwaters, the iron is mostly ferrous bicarbonate which is a colourless substance. When the groundwater with higher concentration of iron is tapped, it quickly oxidizes to ferric state in the form of an insoluble brown substance, ferric hydroxide. So, that field test[7, 8] will be more suitable which is applicable for estimation of total iron. The present paper discusses an easy approach for testing total iron in field.

EXPERIMENTAL

[a] Bathophenanthroline [4,7-diphenyl-1,10-phenanthroline] 2 g
[b] Thioglycolic acid 100 mL

Note: Thioglycolic acid not only acts as solvent but it also reduces iron(III) [if present] almost instantaneously to iron(II), hence total iron can be estimated.



$$2Fe^{3+} + 2HS{-}CH_2{-}COO^- \longrightarrow 2Fe^{2+} + 2H^+ + \begin{array}{l} S{-}CH_2{-}COO^- \\ | \\ S{-}CH_2{-}COO^- \end{array}$$

Method

In 10 mL water sample add 3 drops of reagent mixture. No change in colour is indicative of absence of iron. The light pink to reddish pink colour developed almost instantaneously is indicative of presence of iron in varying concentrations [0.3 to 10 mg/L] which is determined by comparing with provided colour chart having standard colour shades with respect to known concentrations indicated therein. If the concentration of iron is less than 0.3 mg/L then pink colour develops after 5–10 min which can be rejected.

Colour Chart

Standard ferrous ammonium sulphate [AR] solution was prepared in different dilutions ranging from 0.3 to 10.0 mg/L. The colours developed at various concentrations were taken as reference colours in the colour chart (Table-1).

TABLE-1
MATCH FOR COLOUR PRODUCED WITH VARYING CONCENTRATION OF Fe(II)

Concentration of Fe(II) (mg/L)	Colours developed (almost instantaneously)
10.0	Dark red
5.0	Fire red
3.0	Red
2.0	Pinkish red
1.0	Dark Pink
0.5	Pink
0.3	Light pink
< 0.3	Very light pink (after 5 min)

Estimation of iron

Standard iron solutions were made from ferrous ammonium sulphate and estimation of ferrous iron and total iron were done using atomic absorption spectrophotometric determination.

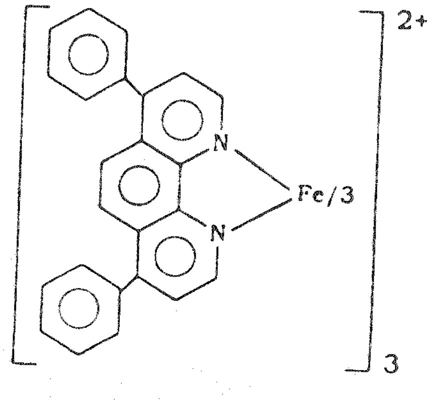
RESULTS AND DISCUSSION

The methods for estimation of iron in water involve the use of different reagents including potassium thiocyanate, α,α′-dipyridyl, thioglycolic acid, dimethylglyoxime, 1,10-phenanthroline and bathophenanthroline. Laboratory studies on these reagents were conducted in view of their adaptability for field determinations, stability, ease and sensitivity of test and nature of complexes formed. The standard reagents used in the laboratory are not suitable for field application due to their complexity, instability and limitation of testing either ferrous or ferric ions. It has been concluded that the bathophenanthroline reagent is more suitable because it is a much more sensitive reagent than even 1,10-phenanthroline (Table-2).

TABLE-2
STANDARD REAGENTS AND RAPID FIELD TEST REAGENT USED FOR TESTING IRON IN WATER

Characteristics	Reagents					
	Potassium thiocyanate	Dipyridyl	Thioglycollic acid	Dimethyl glyoxime	1,10-phenanthroline	Bathophenanthroline
Medium	Mineral acid	Alcohol	Ammonia	Alcohol	Water	Thioglycollic acid
No. of ingredients	2	2	2	4	3	2
Sensitivity (mg/L)	0.25	0.03	0.01	0.40	0.001	0.3
Oxidation state of Iron	Fe^{3+}	Fe^{2+}	Fe^{2+} and Fe^{3+}	Fe^{2+}	Fe^{2+}	Fe^{2+} and Fe^{3+}
Nature of complex	Red, unstable	Dark red, stable	Red-violet, unstable	Red, unstable	Pink to orange red, stable	Light pink to reddish pink, stable
Remark	Liquid phase, short life	Soluble in alcohol, short life	Unpleasant odour, liquid phase	Soluble in alcohol, short life	Soluble in water, liquid phase	Soluble in water, liquid phase

It is reported that bathophenanthroline is specific for determination of dissolved iron $[Fe^{2+}]$. Each atom of iron chelates with three molecules of bathophenanthroline to form a reddish pink complex as shown below, which is soluble in acid medium.



Structure

A pH between 2.0 to 3.5 favours rapid colour development and colours developed are quite stable. The interfering substances in this test are strong oxidizing agents like cyanide, nitrite, free chlorine, phosphate, chromium, zinc, cobalt, copper, nickel, bismuth, cadmium, mercury, molybdate and silver which are usually absent in normal untreated water.

The test has been modified for field application by selecting the amount of reagent.

Water samples having total dissolved solids up to 3000 mg/L and pH up to 9.2 (maximum permissible limits of TDS and pH in drinking water) have responded well to this test. The final pH of the test solution has been found to range between 3.20 to 3.40 as given in Table-3.

TABLE-3

FUNCTION OF IRON-REAGENT UPON TDS AND pH OF WATERS

Water Quality		pH	
Constituents	Range	Initial	Final*
TDS	3000 mg/L	7.5	3.20
$Ca^{2+} + Mg^{2+}$	15-300 mg/L	8.2	3.20
$Na^{2+} + K^{+}$	5–250 mg/L	8.5	3.30
Cl^{-}	25–1200 mg/L	9.2	3.30
$CO_3^{2-} + HCO_3^{-}$	50–500 mg/L	9.5	3.40
SO_4^{2-}	35–700 mg/L	—	—

*After adding field test reagent

The colour produced in every set of tests is stable. The advantages associated with new modified field test are:

1. Thioglycolic acid makes the water acidic (pH 2.9–3.5) which is the most favourable condition for the formation of complex and the colour of the metal complex formed is characteristic and sharp that it can form basis for a reliable spot test for detection of iron.
2. The thioglycolic acid easily reduces Fe^{3+} to Fe^{2+} and thus favours dissolution of total iron.
3. The reducing nature of thioglycolic acid also controls to a little extent the interference caused by oxidizing agents (if present).
4. Water samples having total dissolved solids up to 3000 mg/L have responded well to this test.

Comparative studies of standard methods (atomic absorption spectrometric) and rapid field test undertaken on standard iron solutions have shown (Table-4) that the visual colour test exhibited by this method is quite reliable under field conditions.

Laboratory and field trials on this reagent have been extensively conducted during the last one year.

TABLE-4
COMPARATIVE STUDIES OF RAPID FIELD TEST AND STANDARD METHODS

Method	Concentration of iron (mg/L)					
	<0.3	0.3	0.5	1.0	5.0	10.0
Atomic absorption spectrometric method	—	0.296	0.502	1.012	5.002	10.004
Rapid field method	faint pink after 5 min.	distinct light pink	Pink	dark pink	reddish pink	dark reddish pink

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