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Analysis of Ions of Iron in Water Samples Using Titrimetry and Potentiometric Techniques

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Abstract: Titrimetic and potentiometric methods were employed for the analysis of the ions of iron (ferrous and ferric) in natural water samples. The method for the determination of iron employs a simple acidification step in order to decompose iron hydroxide and iron-complexes into free iron, Fe(III) and Fe(II). The amounts of free iron were detected titrimetrically and electro analytically .Titrimetric analysis was done using using potassium dichromate wherein an oxidation reduction reaction was involved. Indicator used was diphenyl amine. The electro analytical technique used was potentiometry. Potentiometric analysis did not require the use of any indicators.It was concluded from the present study that potentiometric analysis was more accurate and less time consuming and the results obtained were reproducible.

Keywords: *Titrimetry, potentiometry.*

I. INTRODUCTION

The safety of drinking water is of utmost concern. World health oganisation has assigned well defined standards for drinking water purity. U.S federal regulations, limit the amount of iron to less than 3 ppm in municipal drinking water. Iron is toxic at high concentrations and acts as a surrogate for other heavy metals whose presence in drinking water poses a danger to public health.

Iron has two means of infiltrating well water, seepage and corrosion. Water in the form of rain travels from the ground's surface and through the soil to become part of a water supply. If the soil contains iron, the iron can dissolve into the water and travel with it. Exposure to a combination of water and oxygen causes iron to deteriorate; the casings and pipes of a well water supply have a passing acquaintance with both factors. If the casings and pipes contain iron, the acquaintance leads to this deterioration. Rust, the natural by-product of iron corrosion, flakes off the well's components and into the water traveling from the well to our taps.(1,2).

Human bodies require iron but heavy doses are toxic. The Environmental Protection Agency considers iron in well water as a secondary contaminant, which means it does not have a direct impact on health. The Secondary Maximum Contaminant Level set out by the EPA is 0.3 milligrams per liter, but this is merely a guideline and not a federal standard. Iron causes costly damage and other issues when present in high concentrations. (3-5).

II. METHODOLOGY

The method for determination of iron involved a simple acidification step in order to decompose iron hydroxide and iron complexes into free iron Fe3+ and Fe2+. The amounts of iron were determined titrimetrically and electroanalytically.

Titrimetry :

Ferrous iron in the sample waters is estimated by titrating against standard $K_2Cr_2O_7$ solution in acid medium using diphenyl amine as the indicator . In order to estimate the ferric iron , all of it , is first reduced to the ferrous state with the help of stannous chloride.

$$2Fe^{3+}_{(aq)} + Sn^{2+}_{(aq)} \text{ ---------- } 2Fe^{2+}_{(aq)} + Sn^{4+}_{(aq)}$$

While reducing the ferric iron , a slight excess of stannous chloride is added to ensue complete reduction .The excess unreacted $SnCl_2$ is removed by the addition of mercuric chloride which oxidizes stannous chloride to stanic chloride.

$$SnCl_2 + 2HgCl_2 \text{ ---------- } SnCl_4 + Hg_2Cl_2$$

Since mercurous chloride is not acted upon by the oxidants used for the titration it is allowed to remain in the solution . After reduction , the solution containing the ferrous iron present originally and the ferrous iron obtained by the reduction of ferric iron is titrated against $K_2Cr_2O_7$ solution and the total amount of Fe^{2+} iron is calculated. Amount of Fe^{3+} iron = Amount of total Fe^{2+} iron –amount of Fe^{2+} iron originally present.

Preparation of standard $K_2Cr_2O_7$ solution :

About 0.63 gm of a pure sample of $K_2Cr_2O_7$ was taken into a 100 ml standard flask and dissolved it in minimum amount of distilled water and then made up to 100 ml. The flask was shaken thoroughly for uniform concentration with molarity 0.022. Amount of Fe^{3+} iron = Amount of total Fe^{2+} iron –amount of Fe^{2+} iron originally present.

Estimation of Ferrous iron :

The sample water containing the ions of iron was titrated against potassium dichromate in the presence of acid mixture (H_2SO_4 and H_3PO_4) . The indicator used was diphenyl amine. The results are tabulated in Table-1 and the amount of ferrous ion was calculated.

Estimation of ferric iron:

Reduction of ferrous to ferric iron

5 ml of conc. HCl was added to the sample water and the contents were heated to boiling. stannous chloride was added dropwise till the yellow colour due to Fe^{3+} ion just disappeared. The solution was cooled and 10 ml of a saturated solution of mercuric chloride was added with stirring to remove excess of $SnCl_2$. A silky white precipitate of Hg_2Cl_2 was formed upon the addition of $HgCl_2$. The solution was discarded when a black or grey or no precipitate was formed and the process was repeated. The sample water was now titrated against potassium dichromate in the presence of acid mixture (H_2SO_4 and H_3PO_4)diphenyl amine indicator. The results are shown in Table-2

Electro analytical analysis- Potentiometry

Phenomenon underlying the method was electrode potential differences of the cell and EMF was measured . The electrodes used were saturated calomel electrode and quinhydrone electrodes. Water samples were titrated against standard

potassium dichromate taken in a burette and the results are tabulated in table-3 and 4. EMF values were noted and a graph of ΔE/ΔV vs. volume of potassium dichromate (Figures -1 and 2) were plotted and equivalence point was noted from the graphical plots.

III. RESULTS AND DISCUSSION

Titrimetry:

Estimation of ferrous iron:

Table 1

S.No.	Vol. of FAS	Burette reading Initial	Final	Volume of $K_2Cr_2O_7$run down
1	20	0	3.2	3.2

Estimation of ferric iron:

Table 2

S.No.	Vol. of mixture	Burette reading Initial	Final	Volume of $K_2Cr_2O_7$run down
1	20	0	8.8	8.8

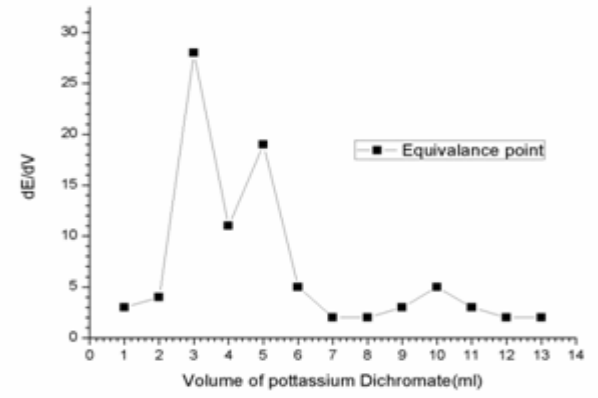
Instrumental Analysis

Graphs ofΔE/ΔV Vs. Volume of potassium dichromate in potentiometry (Figure-1 and 2). The data is as shown in Tables 3 and 4 for ferrous and ferric ions respectively.

Estimation of ferrous

Table 3Δ

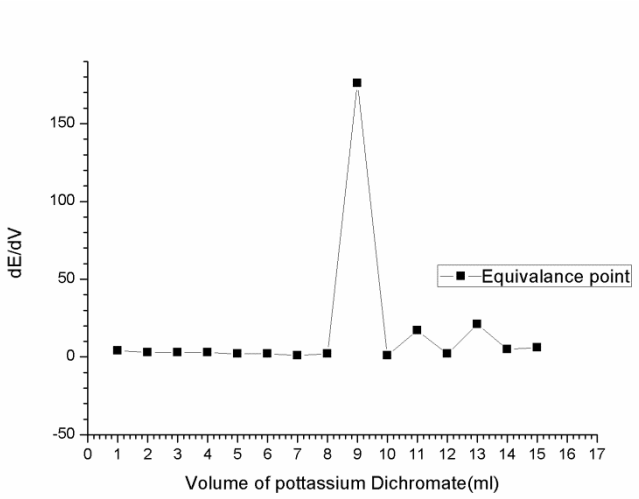
S.No.	ΔE/ΔV	Volume of pottassium Dichromate
1	2	O
2	3	1
3	4	2
4	4	3
5	28	4
6	19	5
7	5	6
8	2	7
9	2	8
10	3	9
11	5	10
12	3	11
13	2	12
14	2	13



Estimation of ferric

Table 4

S.No.	ΔE/ΔV	Volume of pottassium Dichromate
1	4	O
2	4	1
3	3	2
4	3	3
5	3	4
6	2	5
7	2	6
8	1	7
9	2	8
10	176	9
11	1	10
12	17	11
13	2	12
14	21	13
15	5	14
16	6	15



Estimation of ferrous:

$K_2Cr_2O_7$ FAS

M_1=0.022 M_2=?

V_1= 3.2 V_2 = 20

n_1= 1 n_2=6

$M_2=(M_1V_1/n_1)*(n_2/V_2)$= 0.021

Amount of Fe^{2+} = $W_1 = M_2 * 56/10$=0.118 gm/100 ml

Estimation of ferric:

$K_2Cr_2O_7$ FAS (Total Fe^{2+})

M_1=0.022 M_3=?

V_1= 8.8 V_3 = 20

n_1= 1 n_3=6

$M_3=(M_1V_1/n_1)*(n_3/V_3)$=0.058

Amount of Fe^{2+} =$W_1 = M_2*56/10$=0.118.gm/100 ml

Amount of total Fe^{2+} $=W_2=M_3*56/10= 0.325$ gm/100ml

Amount of Fe^{3+} $=W_2-W_1 =0.207$ gm/100 ml

CONCLUSION

The results obtained for volumetric, potentiometric analyses were found to be almost the same. The analysis an the results by electroanalytical technique were found to be more accurate, less time consuming than volumetric analysis. Potentiometric titrations are based on standard electrode potential change observed through potentiometer . Whilst direct titrations are based on physical observation on color change (6-8). The detection of the endpoint can be noted significantly by a drastic change in potential (in potentiometry) . So both precision and accuracy could be achieved. But in the case of direct titration using an indicator, change in the color is the criteria and the observation of color change can vary from one person to other and so both precision as well as accuracy cannot be achieved. For instance using di phenyl amine indicator, the color change observation may significantly vary from person to person. Electro analytical analyses eliminate any indicator blank error and pinpoint accuracy over volumetric titration was obtained (9,10).Hence it could be concluded that electro analytical techniques are better methods.

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References

[1] Benjamin, Mark. Water Chemistry. New York:McGraw-Hill, 2002.

[2] Adsorption of natural dissolved organic matter at the oxide/water interface James A. Davis.Swiss Federal Institute for Water, Resources and Water Pollution Control, CH-8600 Dubendorf, Switzerland.

[3] Adsorption and desorption of different organic matter fractions on iron oxide. Geochimica et Cosmochimica Acta Volume 59, Issue 2, January 1995, Pages 219–229

[4] Ion-exchanger colorimetry—I: Micro determination of chromium, iron, copper and cobalt in water Kazuhisa Yoshimura. Author links open the author workspace.Hirohiko Waki. Author links open the author workspace.Shigeru Ohashi. Author links open the author workspace. Department of Chemistry, Faculty of Science, Kyushu University, Hakozaki, Higashi-ku, Fukuoka, Japan.

[5] Determination of total and dissolved amount of iron in water samples using catalytic spectrophotometric flow injection analysis S. Lunvongsa, M. Oshima, S. Motomizu Talanta Volume 68, Issue 3, 15 January 2006, Pages 969–973.

[6] Solubilities of salts and kinetics of reaction between hydroxide ions and iron(II)–di-imine complexes in water–methanol mixtures. Derivation of single-ion transfer chemical potentials and their application to analysis of solvent effects on kinetic parameters Michael J. Blandamer, John Burgess, Barbara Clark. J. Chem. Soc., Faraday Trans. 1, 1986,82, 1471.

[7] Test procedures in accordance to ISO:3025 (part – 23) Reaffirmed 2003.

[8] AWWA, WEF, APHA, 1998, Standard Methods for the Examination of Water and Wastewater (Methods: 4500 B. Electrometric Method; 2320 B. Titration Method) Sawyer, C.N., McCarty, P.L., and Parkin, G.F. 2000.

[9] Chemistry for Environmental Engineering 4th Edition. Tata McGraw-Hill Publishing Company Limited.

[10] 1963. Laboratory studies on mesophilic and thermophilic anaerobic sludge digestion. J.WaterPollut. Control Fed.35:11