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A SIMPLE, PRECISE AND HIGHLY EFFICIENT ANALYTICAL METHOD FOR THE ESTIMATION OF TRACE AMOUNT OF IRON PRESENT IN BISPHENOLS

N. D. SHINDE*, L. S. PATIL, D. V. JADHAV, V. S. SURYAWANSHI, O. B. PAWAR and S. S. GAIKWAD

Department of Chemistry, Shri Chhatrapati Shivaji College, OMERGA – 413606 (M.S.) INDIA

ABSTRACT

A simple spectrophotometric method for the estimation of iron from Bisphenol was developed. The proposed method is based on a coupled two-step redox and complexation reaction. In the first step, iron present in bisphenol in higher oxidation state Fe (III) is converted into Fe (II) ion by reduction with hydroxylamine hydrochloride. Subsequently, Fe (II) is complexed with Ferrozine reagent.

Key words: Bisphenol, Ferrozine, Iron (II), Ashing, UV-Vis spectrophotometer.

INTRODUCTION

Bisphenol is an important raw material for polymer industries[1]. While referring literature of bisphenol and its applications, it was found that iron is an agent that changes the color of bisphenol, which is the most important parameter characterizing the quality of bisphenol[2]. For the estimation of iron, literature review reveals that various compounds containing the ferroin group have been synthesized[3] and majority of group demonstrate the ability to form complexes with the ferrous ion but most of these complexes are unstable under normal conditions, weakly colored as well as formed over a very narrow pH range. However, some of these compounds form stable and intensely colored species with the ferrous ion. Examples of such compounds are 1,10-phenanthroline[4], 4,7-diphenyl-1,10-phenolthroline[5], 2,2'-bipyridine[6], 2,6-bis 2-pyridyl)-pyridine[6], phenyl 2-pyrodyl ketoxime[7] and 2,4,6-tris (2-pyridyl)-1,3,5-triazine[8]. Many of these compounds are the product of difficult organic syntheses and hence, expensive in cost[9]. In 1970, L. L. Stookey reported

*Author for correspondence; E-mail: chem_scsco@rediffmail.com

application of ferrozine as a reagent for iron estimation up to nanomolar concentrations[10]. The two major advantages of the use of ferrozine are its sensitivity and low cost[10,11].

UV-Vis spectrophotometry is the most widely used technique in analysis because it is simple, economic and easily available to most quality control laboratories. In the present work, we developed a simple, sensitive and cost effective spectrophotometric method for the reliable estimation of iron from bisphenol.

EXPERIMENTAL

Shimadzu double beam UV-Vis Spectrophotometer-1601 with matched quartz cells were used for the present investigation. All the chemicals used in this investigation were of A. R. grade and double distilled water was used for preparation of solutions.

Reagent preparation

Hydroxylamine hydrochloride (Aldrich, 25% w/v): 25 g of hydroxylamine hydrochloride was accurately weighed and dissolved in 100 mL of distilled water.

Sodium acetate trihydrate solution (Aldrich, 1N): Dissolved 136.08 g of sodium acetate trihydrate in 500 mL distilled water.

Ferrozine (Sigma, 0.1% w/v): 1 g of Ferrozine was accurately weighed and dissolved in distilled water and diluted it to 1000 mL with distilled water.

Standard preparation

Stock solution of Fe (III) (1000 ppm): Dissolved 0.8634 g of ferric ammonium sulphate in about 50 mL distilled water with addition of 2-3 drops of HCl then diluted it to 100 mL with distilled water in a 100 mL volumetric flask.

Working standard solution of Fe (III) (100 ppm): 10 mL stock solution was further diluted with distilled water in a 100 mL volumetric flask.

Procedure for calibration curve

Aliquots of standard solution (100 ppm) 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 1.0, 1.5 and 2.0 mL transferred in to a series of 100 mL volumetric flask. 2 mL HCl was added to each flask then 5 mL hydroxylamine hydrochloride solution was added to all flasks. Then 50 mL

sodium acetate solution was added to each flask and finally 2 mL ferrozine reagent was added to all flasks and let set for 30 min. and final volume was made up to 100 mL with distilled water.

The absorbance was measured at λ_{max} 565 nm against corresponding reagent blanks. The amount of Fe (II) in sample was estimated from corresponding calibration graph.

Sample preparation

5 g of 4, 4'-(cyclohexane-1,1diyl) bis (2-methylphenol) was accurately weighed into quartz crucible and added 3-4 drops of H_2SO_4. Placed the quartz crucible on hot plate and increased slowly temp to char the sample. Then placed quartz crucible in muffle furnace and increased slowly temperature up to 800^oC and kept for 6 h to convert the sample into ash. After cooling the muffles furnace, 1 mL HCl was added carefully in crucible and transferred into 100 mL volumetric flask then added 5 mL hydroxylamine hydrochloride solution and 20 mL sodium acetate solution. Finally 5 mL ferrozine solution was added and let set for 30 min. and final volume was made up to 100 mL with distilled water. The absorbance was measured at λ_{max} 565 nm.

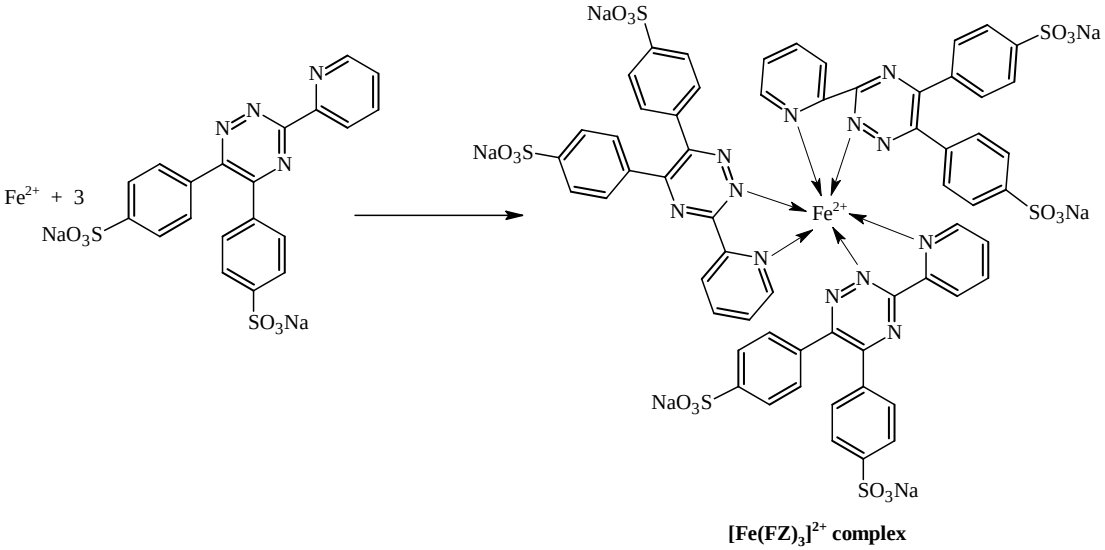
RESULTS AND DISCUSSION

The proposed method is based on the coupled redox-complexation reaction. In the first (redox) step of the reaction (**Scheme 1**), hydroxylamine hydrochloride reduces Fe (III) to Fe (II). This reaction is carried out in the presence of sodium acetate, which prevents precipitation of easily hydrolyzed cations. In the second step of the reaction (**Scheme 2**), *in situ* formed Fe (II) is immediately complexed by 3 molecules of ferrozine to form the dark magenta colored, highly stable $[Fe(FZ)_3]^{2+}$ complex which absorbs light at λ_{max} 565 nm.

The intense color allows trace analysis to be done on these ions in solution using light absorption measurement. The ferrous ion Fe (II) become highly absorbing to visible light when it forms a complex with organic chelator Ferrozine. This is very stable complex, is dark magenta and can be spectrophotometrically detected even at very low amount of iron present in bisphenols. The ashing method proposed in this work proves quite adequate. Linearity and range of this method were confirmed by concentration range and correlation coefficient was within limits. This method can be applied industrial purpose for analysis of bisphenols.

$$2HONH_2.HCl + 2\,Fe^{3+} \rightleftharpoons 4\,H^+ + 2\,H_2O + N_2 + 2\,Fe^{2+} + 2\,Cl^-$$

Scheme 1



$[Fe(FZ)_3]^{2+}$ complex

Scheme 2: Formation of $[Fe(FZ)_3]^{2+}$ complex.

Table 1: Calibration curve line for determination of Fe (II) in the form of complex with Ferrozine

S. No.	Std. concentration of Fe (ppm)	Absorbance
1	0.10	0.05
2	0.20	0.09
3	0.30	0.14
4	0.40	0.19
5	0.50	0.23
6	0.60	0.27
7	0.70	0.32
8	1.00	0.46
9	1.50	0.68
10	2.00	0.92

Correlation coefficient = $r^2 = 0.99976$.
Acceptance criteria = should not be less than 0.999.

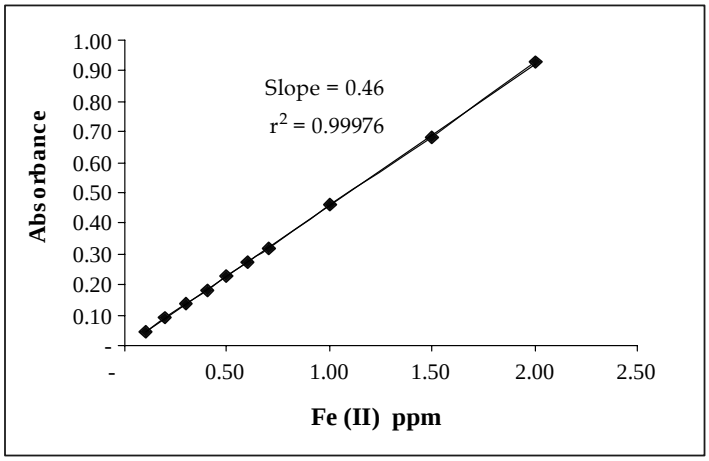


Fig. 1: The calibration line for determination of Fe (II) in the form of a complex with Ferrozine

Table 2.

S. No.	Description	Absorbance
1	Sample	0.0162
2	Blank	0.005

Table 3: Calculation table for determination of r^2 value

X value	Y value	—	—	—	—	—	—
0.1	0.05	-0.63	-0.2855	0.179865		0.3969	0.08151
0.2	0.09	-0.53	-0.2455	0.130115		0.2809	0.06027
0.3	0.14	-0.43	-0.1955	0.084065		0.1849	0.03822
0.4	0.19	-0.33	-0.1455	0.048015		0.1089	0.02117
0.5	0.23	-0.23	-0.1055	0.024265		0.0529	0.01113
0.6	0.27	-0.13	-0.0655	0.008515		0.0169	0.00429
0.7	0.32	-0.03	-0.0155	0.000465		0.0009	0.00024
1.0	0.46	0.27	0.1245	0.033615		0.0729	0.0155
1.5	0.68	0.77	0.3455	0.265265		0.5929	0.11868
2.0	0.92	1.27	0.5845	0.742315		1.6129	0.34164
—	—			**1.5165**		**3.321**	**0.692653**

$$r = \frac{\sum (x - \bar{x})(y - \bar{y})}{\sqrt{\sum (x - \bar{x})^2 (y - \bar{y})^2}}$$

Numerator for r = 1.5165

Denominator for r = 1.516674

r value = 0.99988

r^2 value = 0.99976

Calculation for estimation of iron (ppm) present in bisphenol

$$\text{Slope} = \frac{Y2 - Y1}{X2 - X1}$$

$$\text{Slope} = \frac{0.46 - 0.23}{1.0 - 0.54}$$

Slope = 0.46

$$= \frac{(\text{Sample absorbance} - \text{Blank absorbance}}{\text{Slope of std. Fe curve} \times \text{weight of sample}} \times 100$$

$$= \frac{(0.0162 - 0.005)}{0.46 \times 5} \times 100$$

Fe (ppm) = 0.4869

CONCLUSION

The proposed method for determination of iron present in bisphenol is simple, sensitive, safe, reproducible and accurate and can be used for the routine analysis of bisphenols. The main advantage of this ashing method is useful for determination of trace amount of iron present in solid compound.

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