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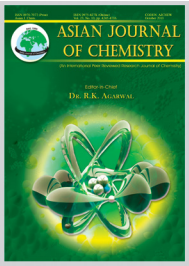

Spectrophotometric Determination of Iron(III) by New Analytical Reagents Derived from Coupling Arylhydroxylamine and Aryldiazonium Salt

O. OMBAKA* and J.M. GICHUMBI

Department of Chemistry, Chuka University College, Post Box 109, Chuka, Kenya

*Corresponding author: E-mail: ombakaochieng@gmail.com

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A highly sensitive and selective spectrophotometric method is developed for the determination of iron(III) in aqueous solution with hydroxytriazenes. In this work, the effect of pH variation, the composition of the complex of Fe^{3+} to hydroxytriazenes, precision, Sandell's sensitivity and interference studies were investigated. This investigation was done under the optimum reaction conditions and other important analytical parameters. Hydroxytriazenes reacts with the Fe^{3+} and forms coloured complexes. The Fe(III) complex is detected at λ_{max} = 410 nm and pH range 3-4 for reagent no(i), 530 nm and 4-5 for reagent no (ii), 410 nm and 4.5 -5.5 for reagent (iii), 396 nm and 3.5-5.0 for reagent (iv), 467 nm and 2.5-3.5 for reagent no.(vi). Beer-Lambert's law is obeyed in the concentration range (1.0-5.0) × 10^{-5} M,(1.5-9.0) × 10^{-5} M, (0.5-4.0) × 10^{-5} M, (0.3-1.8) × 10^{-5} M, (0.6-3.6) × 10^{-5} M, (2-12) × 10^{-5} M for nos. (i), (ii), (iii), (iv), (v), (vi) respectively. The molar ratio of Fe(III) to the hydroxytriazenes was found as 1:3. The limiting concentration for interference for 35 diverse ions are reported. The standard deviation ranges between 0.019 to 0.088. The Sandell's sensitivity in ng/cm^3 ranges between 2.5.579 to 4.189. Reagent nos. (iii), (iv), (v), which were very sensitive were used to determine the level of iron in vegetable samples from Baharini, Nakuru Town and the results obtained were compared with those of atomic absorption spectrophotometer.

Key Words: Hydroxytriazenes, Spectrophotometer, Sandell's sensitivity.

INTRODUCTION

The separation and determination of heavy metal ions in the environmental and biochemical research have been one of the most important topics of analytical chemistry. As compared with the other techniques, spectrophotometry is simple, rapid and less expensive for determination of elements in a variety of samples. Developing highly functional chelating agents which are cheap, sensitive and selective for determination of heavy metals have been a great concern of many analytical chemists. Numerous reagents have been reported for the spectrophotometric determination of iron (III), for example thiocynate and chloromazine[1], *N*-nitro benzohydroxamic acid[2], 9,10-phenanthrenequinone[3,4]. Iron plays an important role in physiology[5], it belongs to the group of so called bi-elements-metals which are necessary for normal development of the living organisms. Iron plays a significant role in biological processes *e.g.*, it takes part in the synthesis of haemoglobin, myoglobin and iron porphyrin enzymes. Its deficiency causes anemia and next atrophy of mucous membranes as a result of disturbed absorption of nutrients. Too much iron, or iron overload, is not so common nor is it so readily treated. Primary haemochromatosis results from a genetic defect leading to enhanced iron uptake. Secondary haemochromatosis occurs because iron has accumulated in the body as a result of the extensive blood transfusions required to ameliorate the effects of other genetic defects, for example those which lead to thalassemia.

This work aimed to develop a highly sensitive, selective and efficient and cheap spectrophotometric method for iron(III) determination, based on the formation of coloured complexes, which were formed by the reaction of iron(III)with hydroxy-triazenes. Various factors influence the sensitivity of the proposed method such as wavelength, pH. Effect of foreign ions and ranges of applicability of the Beer-Lambert's law in the determination of the iron(III) are also included. The method has been applied for determination of iron in vegetable samples from Baharini and Nakuru Town of Kenya which the owner uses sewage water for irrigation purposes.

EXPERIMENTAL

Solutions for spectrophotometric determination

Standard stock solution of iron(III): A 1.0 × 10^{-2} M stock solution of iron(III) was prepared by dissolving required quantity of A.R. grade ferric nitrate nonahydrate in double distilled deionized water. To prevent hydrolysis a few drops of concentrated nitric acid were added to the solution. The

solution was then standardized using 1.0×10^{-2} M EDTA solution and sulphosalicylic acid as an indicator. Lower concentrations were prepared by diluting stock solutions.

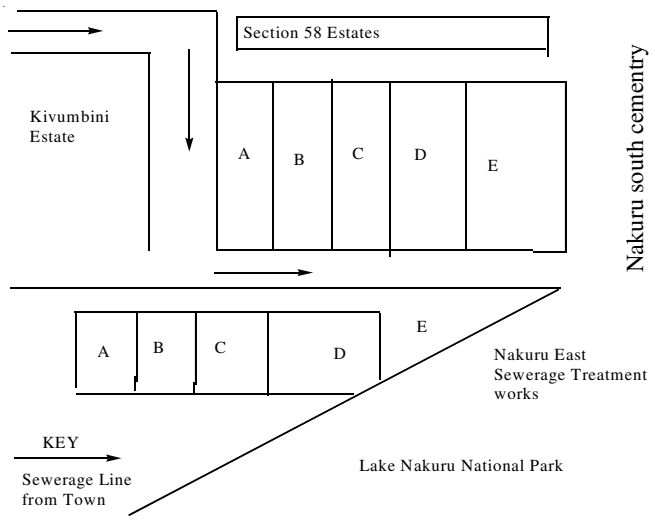
Reagent solutions: Stock solution of each hydroxytriazenes (1.0×10^{-2} M) was prepared by dissolving the required quantity of it in ethanol (reagent nos. (iii), (vi) or acetone for reagent nos. (i), (ii), (iv), (v). Lower concentrations were prepared by dilution using either acetone or ethanol depending on the solubility of hydroxytriazenes.

Solutions for pH adjustments: A 1.0 % *tris*-buffer solution was prepared by dissolving the required quantity of *tris*-buffer in minimum quantity of double distilled deionized water and then diluted to 100 mL using either acetone or ethanol depending on the solubility of hydroxytriazenes. 1 % perchloric acid solution was also prepared by measuring 1 mL perchloric acid and diluted to 100 mL using either acetone or ethanol depending on the solubility of hydroxytriazenes.

Solution for atomic absorption spectrophotometric determination

Standard solutions: Standard solutions for iron were prepared from 1000 ppm spectro econ stock solution by serial dilution to the range of standards as recommended by the manufacturers of AAS CTA 200 model.

Design and location of the study area: The area of study is Baharini estate, Bondeni location, municipality division, Nakuru town, Kenya. It borders Kivumbini estate to the west, Nakuru South Cemetery to the East, Nakuru East Sewage treatment works, Lake Nakuru National Park to the south and section 58 to the North.



Sampling of vegetable and mineralistion[6-8]: The leave samples were taken at five different sampling points. The sampling was carried out at the mid day. The samples were collected in paper bags in order to reduce decomposition prior for analysis. The samples were then transported to the laboratory whereby, they were subjected to drying in an oven set at 60 ºC for 48 h. Thereafter, grinding was carried out using a mortar and pestle until all the material passed a 1 mm mesh. About 25 g of the samples from each sampling point was retained by coning and quartering to act as a representative sample.

About 1 g of dry, ground sample was weighed into an acid-washed porcelain basin, which was previously ignited at 550 ºC for 2 h in a muffle furnace. The porcelain containing sample was then placed in a cold muffle furnace and the temperature was allowed to rise slowly to 550 ºC. This temperature (550 ºC) was then maintained for 2 h. The porcelain containing the residue was removed and allowed to cool. After cooling, 10 mL of 50 % HCl was added followed by covering with a watch glass and thereafter, heated on a steam bath for 15 min. Then 1 mL of conc. HNO_3 was added and evaporated to dryness and heating continued for 1 h. Thereafter 2 mL of 50 % HCl was added followed by swirling in order to dissolve the residue. The solution was then diluted to 10 mL using freshly doubly distilled deionized water and warmed to complete dissolution. The solution was filtered through a No. 44 filter paper into a 100 mL volumetric flask and diluted to volume. This was taken as sample stock solution. The blank determination was carried out the same way. Both the sample and blank solutions was diluted 20 times to obtain the solutions used in spectrophotometer and atomic absorption spectrophotometer.

Synthesis of the hydroxytriazenes: The synthesis of the hydroxytriazenes involves coupling of aryldiazonium salt with aryl or alkyl hydroxylamine[9]. The pH measurements were carried out using a systronics pH meter 324. The investigation was carried out at the room temperature by systronics UV-VIS. Spectrophotometer 108 using 1 cm quartz cells. Atomic absorption spectrophotometer, model CTA 2000 was also employed.

Spectrophotometric procedure[10]

Determination of working wavelength: 1 mL of 1×10^{-4} M Fe^{3+} was taken into a 10 mL volumetric flask. Then 5 mL of 1×10^{-4} M hydroxytriazenes was added. The solution was topped to the mark using double distilled deionized water. The absorbance was then determined in wavelength region against ethanol or acetone was measured. The plot of absorbance against wavelength was then done on the same graph.

Determination of pH range: A set of solutions having concentrations of Fe^{3+} and hydroxytriazenes were prepared in such a manner that, the ratio of [M]:[R] was 1:5. The pH of these solutions was adjusted in such a manner that, each solution was having the pH different from others. This was done using 1 % *tris*-buffer and 1 % perchloric acid. Then the absorbance of each solution was measured against reagent blank. The absorbance obtained was then plotted against their corresponding pH.

Molar composition of iron(III) complex with each of the hydroxytriazenes: Three different methods, namely Job's method, mole ratio method and slope ratio method of Harvey and Manning were used for the determination of molar composition of metal: reagent.

Validity of Lambert-Beer's law: Under corresponding optimum conditions of pH, solvent and iron(III) to reagent ratio, the validity of Beer-Lambert's law was studied. The results of absorbance obtained were plotted against the corresponding concentration of iron(III). The calibration curve obtained was used to determine the concentration of iron in sample solution after obtaining the absorbance of the sample.

TABLE-1
SPECTROPHOTOMETRIC DETERMINATION OF IRON(III) WITH HYDROXYTRIAZENES

Reagent no.	Colour and shape of crystals	Solvent for crystallization	m.p. (°C)	Composition of the complex Fe: R	Colour of the complex	λ_{max} (nm)	Working wavelength (nm)	Optimum pH range	Beer-Lambert range × 10^5	Stability constant of complex	Free energy (kcal/mol) of formation of the complex at 25°C	Sandell's Sensitivity ng/cm^2	Precision	
													Fe taken (ppm)	Standard deviation in ppm for ten determinations
(i)	Pale yellow shining needle	Acetone	148	1:3	Blue	397	410	3.0-4.0	1.0-5.0	3.036×10^{15}	-21.113	9.466	2.792	0.032
(ii)	Light yellow powder	Acetone	152	1:3	Green	530	530	4.0-5.0	1.5-9.0	3.903×10^{14}	-20.143	17.272	4.189	0.048
(iii)	Light yellow shining needle	Ethanol	180	1:3	Blue	400	410	4.5-5.5	0.5-4.0	4.303×10^{15}	-21.319	8.865	1.955	0.026
(Iv)	Light yellow shining needle	Acetone	155	1:3	Blue	389	396	3.5-5.0	0.3-1.8	8.117×10^{16}	-23.059	4.189	1.005	0.019
(v)	Light yellow shining needle	Acetone	136	1:3	Blue	394	399	4.0-5.0	0.6-3.6	6.464×10^{15}	-21.560	6.981	1.675	0.029
(vi)	Light yellow shining needle	Ethanol	112	1:3	Green	467	467	2.5-3.5	2.9-12.0	2.623×10^{14}	-19.662	25.579	6.702	0.088

Precision studies: This was carried out by measuring the absorbance of ten solutions. Each solution contained the same concentration of iron(III) and the reagent. The absorbance was measured against reagent blank under optimum conditions of pH, solvent and iron(III) to reagent ratio. On the basis of their absorbance values, standard deviation was determined.

Interference studies[10]: Iron(III) was determined with each reagent under its optimum conditions of pH, solvent and iron(III) to reagent ratio, in the presence of equivalent amounts, five and ten fold excess of 35 diverse ions.

Procedure for atomic absorption spectrophotometric analysis: The instrument was put on and left to warm up for about 0.5 h. The following optimum operating conditions were set before any measurements were made.

Filament	Iron
Wavelength (nm)	248.3
Bandwidth (nm)	1
Filament	Iron
Lamp current (Ma)	8
Lamp gain	4
EHT volts	420
Oxidant-fuel flow	Air
Burner height (cm)	7
Sensitivity (mg/mL)	0.06
Detection limit (mg/mL)	0.003
Calibration range (mg/mL)	0.1-10

After the optimization of the above conditions, aspiration of the sample, series of standard containing the sample having the same concentration of iron were aspirated hence standard addition method was used.

Hydroxytriazenes used as a complexing reagent in spectrophotometric determination of iron(III): (i) 3-Hydroxy-3-*m*-tolyl-1-*p*-chlorophenyltriazene; (ii) 3-Hydroxy-3-*m*-tolyl-1-*m*-nitrophenyltriazene; (iii) 3-hydroxy-3-*m*-tolyl-1-*p*-sulphonato(sodium salt)phenyl triazenes; (iv) 3-Hydroxy-3-*m*-tolyl-1-*m*-chlorophenyltriazene; (v) 3-Hydroxy-3-*m*-tolyl-1-(3,4-dichlorophenyl)triazenes; (vi) 3-Hydroxy-3-*m*-tolyl-1-*p*-methoxyphenyltriazene.

RESULTS AND DISCUSSION

The spectrum of the complex with each reagent was obtained in the wavelength region 380 to 640 nm against reagent blank. Further, spectrum of reagent was also measured in the same wavelength region against ethanol or acetone. The working wavelength was chosen such that there was maximum difference between the absorbance due to complex and reagent. Table-1 indicates that the working wavelength are close to λ_{max}. This ensures that sensitivity is high, molar absorptivity remains constant and Lambert-Beer's law is obeyed.

The absorbance of a series of solutions containing iron(III) and reagent in the molar ratio 1:5[M]:[R] was measured against a blank at corresponding working wavelengths at various pH values in order to determine the pH range of constant and maximum absorbance. This is necessary in order to obtain high sensitivity and also absorbance should not change due to small changes in pH. The pH of the solution was adjusted using 1 % *tris*-buffer and 1 % perchloric acid solutions. The colour development in pH range was instantaneous, stable for *ca.* 24 h. The data for pH range and type of colour developed have been incorporated in Table-1. The molar composition of iron(III) complex with each of the hydroxytriazenes were carried out under corresponding optimum conditions of pH, solvent and iron(III) to reagent ratio using Job's method, mole ratio method and slope ratio method of Harvey and Manning. The results have been included in Table-1. The ratio of [M]:[R], which was employed during verification of Lambert-Beer's law was obtained from mole ratio curve and should be such that, absorbance remains constant as the concentration of reagent is increasing and this will contribute to Lambert-Beer's law being obeyed.

TABLE-2
DETERMINATION OF IRON (III) IN PRESENCE OF INTERFERENCE SPECIES AT EQUIVALENT AMOUNT

Reagent no.	Working wavelength (nm)	pH range	[Fe] $\times 10^{-5}$ M	Absorbance in absences of interfering species	[Fe]: [R]	Absorbance when interfering anionic species was present						
						Cl^-	Br^-	CH_3COO^-	CO_3^{2-}	PO_4^{3-}	SO_4^{2-}	$C_2O_4^{2-}$
(i)	410	3.0-4.0	4.0	0.598	1:6	0.604	0.594	0.594	0.598	0.568	0.593	0.623
(ii)	530	4.0-5.0	5.0	0.472	1:6	0.476	0.472	0.464	0.478	0.420	0.472	0.194
(iii)	410	4.5-5.5	4.0	0.632	1:6	0.632	0.638	0.632	0.633	0.560	0.635	0.583
(iv)	396	3.5-5.0	0.5	0.401	1:6	0.391	0.398	0.393	0.401	0.303	0.401	0.368
(v)	399	4.0-5.0	2.0	0.360	1:6	0.364	0.363	0.364	0.360	0.305	0.360	0.281
(vi)	467	2.5-3.5	8.0	0.536	1:6	0.540	0.546	0.541	0.541	0.245	0.546	0.389
	Absorbance when interfering anionic species was present											
	I^-	$S_2O_3^{2-}$	NO_2^-	SO_3^{2-}	S^{2-}	HPO_4^{2-}	F^-	NO_3^-	WO_4^{2-}	$Mo_7O_{24}^{6-}$	NH_4^+	Na^+
(i)	0.598	0.596	0.588	0.598	0.588	0.552	0.430	0.599	0.598	0.567	0.598	0.598
(ii)	0.478	0.466	0.472	0.462	0.298	0.388	0.424	0.472	0.424	0.474	0.464	0.464
(iii)	0.635	0.638	0.640	0.637	0.515	0.570	0.362	0.639	0.632	0.723	0.638	0.634
(iv)	0.397	0.399	0.394	0.405	0.349	0.287	0.015	0.408	0.385	0.406	0.397	0.399
(v)	0.359	0.343	0.360	0.358	0.358	0.312	0.203	0.360	0.355	0.365	0.360	0.362
(vi)	0.529	0.543	0.529	0.540	0.542	0.391	0.166	0.542	0.536	0.585	0.538	0.536
	Absorbance when interfering cationic species was present											
	K^+	UO_2^{2+}	Mn^{2+}	Ba^{2+}	Pb^{2+}	Hg^{2+}	Sn^{2+}	Th^{4+}	Cd^{2+}	Mg^{2+}	Ca^{2+}	ZrO^{2+}
(i)	0.588	0.566	0.521	0.590	0.564	0.557	0.559	0.589	0.543	0.588	0.596	0.560
(ii)	0.478	0.464	0.396	0.472	0.470	0.464	0.436	0.466	0.470	0.480	0.462	0.480
(iii)	0.634	0.631	0.623	0.634	0.639	0.638	0.623	0.637	0.623	0.624	0.617	0.791
(iv)	0.402	0.375	0.311	0.391	0.374	0.391	0.399	0.399	0.371	0.407	0.398	0.365
(v)	0.360	0.359	0.361	0.363	0.364	0.361	0.362	0.362	0.357	0.362	0.361	0.354
(vi)	0.541	0.576	0.543	0.542	0.541	0.549	0.540	0.536	0.542	0.536	0.529	0.564
	Absorbance when interfering cationic species was present											
				Co^{2+}	Cu^{2+}	Zn^{2+}	Ni^{2+}					
(i)				0.550	0.678	0.549	0.576					
(ii)				0.448	0.396	0.464	0.444					
(iii)				0.617	0.791	0.624	0.636					
(iv)				0.384	0.648	0.354	0.389					
(v)				0.339	0.376	0.268	0.344					
(vi)				0.514	0.544	0.532	0.546					

-Indicates that ion interfered at lower concentration and hence interference studies at next concentration were not done

The stability constants of iron(III) complexes were determined using Harvey and Manning's method. The results from Table-1 reveals that, the complex formed by reagent (iv) is more stable while that formed by reagent (vi) is the least stable. The free energy of formation of the complex using hydroxytriazenes were calculated and the results indicates that, during formation of the complex between Fe(III) and reagent (iv) high amount of energy is given out while using reagent (vi) produces the least energy.

Sandell's spectrophotometric sensitivity was calculated from the values of molar absorptivity. These values indicate that reagent (iv) is the most sensitive while reagent (vi) is the least sensitive.

The precision studies were carried out in order to check the reliability of the results of iron(III) determination with each of the hydroxytriazenes and the results obtained have been given in Table-1. A careful examination of the table shows a fair replication of the results with the reagent studied. Iron(III) has been determined spectrophotometrically in the presence of 35 diverse ions at equivalent amounts, five fold excess and ten fold excess with each of the hydroxytriazenes under their respective optimum conditions. The results obtained from these studies have been given in Tables 2-4. From these results Cl^-, Br^-, CH_3COO^-, CO_3^{2-}, SO_4^{2-}, I^-, NO_2^-, SO_3^{2-}, NO_3^-, NH_4^+, K^+, Ba^{2+}, Th^{4+}, Mg^{2+},Ca^{2+} do not interfere with the determination of iron(III) even if these species are present in ten fold excess with all the hydroxytriazenes used. PO_4^{3-}, $C_2O_4^{2-}$, HPO_4^{2-}, F^-, Co^{2+}, Cu^{2+} interfered with iron(III) determination with all hydroxytriazenes even at equivalent amounts.

A close examination of these tables of the interference studies reveals that, reagent nos. (ii) is the most selective reagent while reagent nos. (iv) is the least selective reagent.

The estimation of iron in vegetable samples were done spectrophotometrically using three most sensitive hydroxytriazenes and the results obtained were compared with those of atomic absorption spectrometer. The results obtained have been summarized in Table-5. The concentration of iron in the sample was ranging from 883 to 1208 ppm, 832 to 158 ppm, 876 to 1227 ppm and 907 to 1284 ppm for reagent no.s (iii), (iv), (v) and AAS respectively.

These values are in decreasing order, down the stream with the samples from site A having the highest levels of iron. Sampling point A is where the freshly incoming sewage water used for irrigation comes into contact with the vegetables after deviation from the main line. The mean value of iron in the vegetable samples were calculated and found to be 995, 939.4, 981.6 and 1036.2 ppm using reagent nos. (iii), (iv), (v) and AAS respectively. The percentage error for reagent (iii), (iv), (v) in comparison with AAS was -4.07, -9.43, -5.36 respectively. The differences in values using hydroxytriazenes and AAS might have been due to the presence of foreign species, which interfered with the determination of iron.

TABLE-3
DETERMINATION OF IRON(III) IN PRESENCE OF INTERFERING SPECIES AT FIVE FOLD EXCESS

Reagent no.	Working wavelength (nm)	pH range	[Fe] $\times 10^{-5}$ M	Absorbance in absences of interfering species	[Fe]:[R]	Absorbance when interfering anionic species was present						
						Cl^-	Br^-	CH_3COO^-	CO_3^{2-}	PO_4^{3-}	SO_4^{2-}	$C_2O_4^{2-}$
(i)	410	3.0-4.0	4.0	0.598	1:6	0.600	0.589	0.597	0.588	-	0.567	-
(ii)	530	4.0-5.0	5.0	0.472	1:6	0.476	0.482	0.476	0.470	-	0.408	-
(iii)	410	4.5-5.5	4.0	0.632	1:6	0.636	0.630	0.628	0.628	-	0.622	-
(iv)	396	3.5-5.0	0.5	0.401	1:6	0.400	0.361	0.400	0.375	-	0.375	-
(v)	399	4.0-5.0	2.0	0.360	1:6	0.364	0.362	0.365	0.361	-	0.357	-
(vi)	467	2.5-3.5	8.0	0.536	1:6	0.536	0.540	0.543	0.561	-	0.573	-
	Absorbance when interfering anionic species was present											
	I^-	$S_2O_3^{2-}$	NO_2^-	SO_3^{2-}	S^{2-}	HPO_4^{2-}	F^-	NO_3^-	WO_4^{2-}	$Mo_7O_{24}^{6-}$	NH_4^+	Na^+
(i)	0.551	0.589	0.551	0.597	0.472	-	-	0.590	0.561	-	0.599	0.598
(ii)	0.476	0.416	0.404	0.376	-	-	-	0.476	-	0.230	0.474	0.472
(iii)	0.630	0.622	0.638	0.623	-	-	-	0.635	0.620	-	0.636	0.638
(iv)	0.368	0.317	0.374	0.305	-	-	-	0.400	-	0.398	0.391	0.393
(v)	0.355	-	0.355	0.329	0.355	-	-	0.364	0.294	0.420	0.362	0.363
(vi)	0.536	0.546	0.513	0.570	0.545	-	-	0.543	0.544	-	0.543	0.536
	Absorbance when interfering cationic species was present											
	K^+	UO_2^{2+}	Mn^{2+}	Ba^{2+}	Pb^{2+}	Hg^{2+}	Sn^{2+}	Th^{4+}	Cd^{2+}	Mg^{2+}	Ca^{2+}	ZrO^{2+}
(i)	0.598	-	-	0.600	-	-	-	0.590	-	0.590	0.590	-
(ii)	0.466	0.360	-	0.468	0.482	0.470	-	0.464	0.482	0.472	0.478	0.466
(iii)	0.626	0.634	0.619	0.634	0.631	0.624	0.596	0.626	0.617	0.627	0.627	0.624
(iv)	0.401	-	-	0.400	-	0.435	0.350	0.385	-	0.411	0.402	-
(v)	0.360	0.355	0.351	0.364	0.349	0.354	0.336	0.338	0.315	0.363	0.362	0.356
(vi)	0.545	-	0.536	0.544	0.565	0.542	0.474	0.540	0.536	0.545	0.539	-
	Absorbance when interfering cationic species was present											
			Co^{2+}	Cu^{2+}	Zn^{2+}	Ni^{2+}						
(i)			-	-	-	-						
(ii)			-	-	0.424	-						
(iii)			-	-	0.614	0.622						
(iv)			-	-	-	-						
(v)			-	-	-	-						
(vi)			-	0.563	0.535	0.557						

-Indicates that ion interfered at lower concentration and hence interference studies at next concentration were not done

TABLE-4
DETERMINATION OF IRON(III) IN PRESENCE OF INTERFERING SPECIES AT TEN FOLD EXCESS

Reagent no.	Working wavelength (nm)	pH range	[Fe] $\times 10^{-5}$ M	Absorbance in absences of interfering species	[Fe]:[R]	Absorbance when interfering anionic species was present						
						Cl^-	Br^-	CH_3COO^-	CO_3^{2-}	PO_4^{3-}	SO_4^{2-}	$C_2O_4^{2-}$
(i)	410	3.0-4.0	4.0	0.598	1:6	0.603	0.544	0.598	0.586	-	-	-
(ii)	530	4.0-5.0	5.0	0.472	1:6	0.474	0.476	0.476	0.474	-	-	-
(iii)	410	4.5-5.5	4.0	0.632	1:6	0.627	0.630	0.640	0.631	-	0.589	-
(iv)	396	3.5-5.0	0.5	0.401	1:6	0.400	-	0.404	-	-	-	-
(v)	399	4.0-5.0	2.0	0.360	1:6	0.364	0.360	0.364	0.361	-	0.314	-
(vi)	467	2.5-3.5	8.0	0.536	1:6	0.536	0.561	0.546	-	-	-	-
	Absorbance when interfering anionic species was present											
	I^-	$S_2O_3^{2-}$	NO_2^-	SO_3^{2-}	S^{2-}	HPO_4^{2-}	F^-	NO_3^-	WO_4^{2-}	$Mo_7O_{24}^{6-}$	NH_4^+	Na^+
(i)	-	0.610	-	0.623	-	-	-	0.593	-	-	0.600	0.597
(ii)	0.480	-	-	-	-	-	-	0.474	-	-	0.470	0.476
(iii)	0.620	0.646	0.621	-	-	-	-	0.628	-	-	0.626	0.629
(iv)	-	-	-	-	-	-	-	0.406	-	0.414	0.394	0.391
(v)	0.319	-	0.323	-	0.317	-	-	0.359	-	-	0.362	0.365
(vi)	0.558	0.546	-	-	0.564	-	-	0.536	0.533	-	0.544	0.539
	Absorbance when interfering cationic species was present											
	K^+	UO_2^{2+}	Mn^{2+}	Ba^{2+}	Pb^{2+}	Hg^{2+}	Sn^{2+}	Th^{4+}	Cd^{2+}	Mg^{2+}	Ca^{2+}	ZrO^{2+}
(i)	0.592	-	-	0.695	-	-	-	0.563	-	0.563	0.555	-
(ii)	0.476	-	-	0.480	0.450	0.466	-	0.504	0.432	0.472	0.480	0.414
(iii)	0.625	0.624	-	0.629	0.616	0.616	-	0.620	-	0.619	0.616	0.604
(iv)	0.401	-	-	0.393	-	-	-	-	-	0.409	0.375	-
(v)	0.359	0.344	-	0.365	-	0.322	-	-	-	0.365	0.364	0.317
(vi)	0.543	-	0.541	0.545	-	0.545	-	0.546	0.557	0.536	0.535	-
	Absorbance when interfering cationic species was present											
			Co^{2+}	Cu^{2+}	Zn^{2+}	Ni^{2+}						
(i)			-	-	-	-						
(ii)			-	-	-	-						
(iii)			-	-	-	0.613						
(iv)			-	-	-	-						
(v)			-	-	-	-						
(vi)			-	-	0.552	-						

-Indicates that ion interfered at lower concentration and hence interference studies at next concentration were not done

TABLE-5
ESTIMATION OF IRON USING SPECTROPHOTOMETRIC (VISIBLE) AND ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHODS

Reagent nos.	Sampling points concentration (mg/Kg)					Mean	Error (%)	F-test at the 95 % confidence level
	A	B	C	D	E			
(iii)	1208	1042	925	917	883	995	-4.07	1.30
(iv)	1158	979	872	856	832	939.4	-9.43	1.28
(v)	1227	995	911	899	876	981.6	-5.36	1.11
AAS	1284	1080	964	951	907	1037.2	--	--

The values of F-test at the 95 % confidence level for reagent nos.(iii), (iv), (v) were found to be 1.30.1.28,1.11 respectively. The tabulated F-values for V1 = 4 and V2 = 4 is 6.39 hence calculated values is less than this value, it indicates that, there is no significant difference in the precision between visible spectrophotometric method using hydroxytriazenes and that of atomic absorption spectrometric method.

Conclusion

Six hydroxytriazenes are introduced as new reagents for spectrophotomeric determination of iron(III). Two of these are very sensitive and selective and can be used for the determination of iron in the presence of a large number of cations and anions and the results are comparable with those of atomic absorption spectrophotometer. These two hydroxytriazenes can be used to determine iron content in vegetable samples. The ease of preparation over many reported reagents, wide pH range, easy methods (direct method) for spectrophotometric determination of iron(III), better yield are further advantages of hydroxytriazenes.

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