The Eurasia Proceedings of Science, Technology,
Engineering & Mathematics (EPSTEM)
ISSN: 2602-3199

The Eurasia Proceedings of Science, Technology, Engineering & Mathematics (EPSTEM), 2019

Volume 7, Pages 23-32

IConTES 2019: International Conference on Technology, Engineering and Science

Spectrophotometric Determination of Hydrazine Sulphate using Fe(II)-2,2'-Bipyridyl-Application to Various Water Samples

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Abstract: Hydrazine is an inorganic chemical compound with the chemical structure N_2H_4. Hydrazine a colorless flammable liquid compound. Hydrazine is very toxic and unstable compound, also hydrazine is a strong reducing agent. This compound easily ignites in the air, generating a large amount of heat and the composition of nitrogen gas and water. Symptoms of acute (short-term) exposure to high levels of hydrazine may include irritation of the eyes, nose, throat, dizziness, headache and nausea. Acute exposure can also damage the liver, kidneys and central nervous system. Cases of lungs, liver, spleen and thyroid infection in animals exposed to hydrazine have been reported by inhalation. Increased lung injury, nasal cavity, and liver tumors were observed in rodents exposed to hydrazine**.** This work involves the development of a fast and sensitive spectrophotometric method for the determination of trace of hydrazine, the method based on the reduction of ferric ions by hydrazing to ferrous ions, and finally the later reacts with the reagent (2,2'-bipyridyl) at 60°C to form a stable red-pink complex, this complex is soluble in water and gave absorption maxima at 523 nm. Beer's law was agreed with range (50-800) µg of hydrazine per 25 ml (i.e: 2-32 ppm), with a corresponding molar absorptivity of 5.6×10^3 $l.mol^{-1}.cm^{-1}$, Sandell's sensitivity index of 0.02 $\mu g.cm^{-2}$ limit of quantitation of 0.3 µg/ml, limit of defection 0.1 µg/ml , and finally average relative standard deviation of $\pm$1.25%, depend on the concentration level. This method had been applied successfully to determination of hydrazine amount in different samples water (tap water, river water and sea water).

Keywords: Hydrazine, Water samples, Molar absorptivity

Introduction

Hydrazine is an important inorganic chemical compound of formula N_2H_4, a base containing nitrogen, a colorless flammable liquid. Hydrazine is very toxic and unstable, and is a strong reducing agent. This compound easily ignites in the air, generating a large amount of heat and the composition of nitrogen gas and water [1-5].The name hydrazine was coined by Emile Fisher in 1875 and was attempting to produce organic compounds consisting of a single-substitution hydrazine. By 1887 [6], The odore Kirtius produced hydrazine sulfate by treating organic diazides with dilute sulfuric acid; however, he was unable to obtain pure hydrazine, despite the efforts mentioned [7]. Pure anhydrous hydrazine was first prepared by Dutch chemist Lopri de Bruyne in 1895[8]. The first use of hydrazine was during World War II as an element in rocket fuel mixtures[9,10]. Due to the many uses of hydrazine in the various fields mentioned above, a number of researchers have developed different methods for estimating hydrazine[11-15].

Various method were reported in the literatures for the estimation of hydrazine such as fluorescence determination of trace amount of Hydrazine[16,17] Chromatographic methods played an important role in the process of hydrazine estimation [18,19].

Spectrophotometric methods were also widely used for the determination of hydrazine after reacting with different ions [20-22], and corrective methods were used to estimate the said compound [23]. Potentiometric

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methods are also approved and recommended in the determination of hydrazine by the use of selective electrodes for this purpose [24,25].

Practical Part

Devices used

Spectroscopic measurements were carried out by a double beam type spectrometer (JASCO 630 double beam UV-visible spectrophotometer) (Japan) Quartz cells with a light path of 1 cm were used. The pH readings were measured using (HANA Instruments pH 211 microprocesser pH meter) An ANDHR-200 type sensitive balance was used for weighing.

Reagents and chemicals used:

All chemicals and analytical reagents used were highly purified.

Solutions of Substances Used:

***Standard hydrazine sulfate solution (1000 micrograms* / ml)**:This solution was prepared by dissolving (0.1000 g) of hydrazine sulfate in 100 mL distilled water using a volumetric flask and was kept in a sealed dark bottle. - Working hydrazine solution (250µg / ml) This solution was prepared by diluting (25 mL) from the standard solution to (100 mL) with distilled water in a volumetric flask and was kept in a dark vial and remaining stable for 15 days.

Regulated solution (pH 2.9) *Prepare* this solution by dissolving (0.082 g) of sodium acetate at a concentration of (1 × 10^{-2} M) with distilled water and diluting the volume to (100 ml) in a volumetric flask.

Reagent solution 2 **', 2- dipyridyl (1 × 10^{-2} M):** Prepare this solution (1× 10^{-2} molar) by dissolving (0.156 g) of the reagent (supplied by ABCR GmbH & Co.KG) in 2 mL ethanol and then complete the volume to (100 mL) with distilled water in a volumetric flask. for a week.

iron ion solution(III) (1 × 10^{-1} M): *Prepare* this solution by dissolving (4.822 g) ammonium ferric sulfate NH_4Fe (SO_4) 2.12H_2O from Dehaen AG-Riedel in distilled water containing 10 drops of concentrated sulphuric acid and diluting the volume to 100 mL in a volumetric bottle and preserving it. In an opaque bottle.

Interfering solutions (1000 µg / ml): These solutions were prepared by dissolving (0.1 g) of each substance in (100 ml) distilled water using a volumetric vial.

Results and Discussion

250 µ/ mL of hydrazine sulphate in final volume (25 mL) was used for subsequent experiments and the absorption of the solutions was measured against the solution.

Principle of method: The current developed spectral method involves two basic steps: The first step is based on the oxidation-reduction reaction between the hydrazine and the iron ion(III), which is reduced to the iron ion (II) by the hydrazine sulfate compound. The second step involves the interaction of the resulting iron-ion (II) with the ′2,2-dipyridyl reagent, which is a complex red-pink complex that gives the highest absorption at a wavelength of 523 nm.

Study of optimal reaction conditions: The effect of various conditions affecting the absorption of the complex formed by the reaction of the resulting iron ion with the reagent 2, 2-bypridyl in the aqueous solution was studied.

Effect of acid function

For the purpose of determining the optimal pH to obtain the reaction between Fe (II) ion and reagent 2 ', 2-bypyridyl and to form a stable and highly colored absorption complex, solutions were prepared with different pH by adding different volumes of HCl solution at 0.01 M. The sodium acetate solution (0.01 molar) to the reaction mixture and complete the volume to (25 mL) with distilled water. Absorption measurements were made for each solution versus the formal solution at a wavelength of 523 nm. The pH of each solution was measured as shown in Table 1. Previous same but using lotion Sodium acetate at a concentration of (0.01 molar) instead of hydrochloric acid and the results are recorded in Table 2.

Table1. Effect of acid addition on the absorption of the complex formed

ml of 0.01 M HCl solution	Absorbance		pH
	Sample vs. blank	Blank vs. water	
0	0.5560	0.1203	2.9
1	0.3371	0.1237	2.85
1.5	0.2590	0.1246	2.83
2	0.2541	0.1247	2.81
3	0.2465	0.1182	2.76
4	0.26866	0.1167	2.73
5	0.2414	0.1248	2.68

It is noted in Table 1 that the addition of acid led to a decrease in the absorption value of the complex formed and therefore was excluded in addition in subsequent experiments. The following table shows the effect of adding different amounts of sodium acetate to the reaction mixture.

Table 2. Effect of sodium acetate addition on complex absorption

ml of 0.01 M CH_3COONa solution	Absorbance		pH
	Sample vs. blank	Blank vs. water	
1	0.5943	0.0901	2.9
2	0.5745	0.0945	2.9
3	0.5598	0.0957	2.92
4	0.5560	0.0884	2.92
5	0.6113	0.1030	2.95
6	0.6438	0.1106	2.95
7	0.6764	0.1066	2.98

It is noted from Table 2 that the addition of sodium acetate gives a good absorption and better than hydrochloric acid and the same solution pH.

To control and preserve the pH at 2.9 for the reaction mixture solution, a number of different structured solutions with an pH of 2.9 were prepared and their effect on absorbance were studied: Glycine-HCl (B_2), KHphthalate-HC l(B_1),Citric acid-NaOH(B_3), Formic acid-NaOH(B_4).

Table 3. Effect of structured solutions on reaction mixture

ml of buffer solution	Absorbance /ml of buffer solution added			
	B_1	B_2	B_3	B_4
1	0.3327	0.3359	0.0864	0.3303
2	0.3076	0.3772	0.0587	0.2848
3	0.2803	0.3733	0.0031	0.2748
5	0.2319	0.3105	-0.1824	0.3305
7	0.2217	0.3309	-0.1050	0.3560
9	0.2213	0.3332	-0.1018	0.3453
Final pH	(3.0-2.8)	(3.0-2.8)	(3.04-2.89)	(3.0-2.8)

Effect of Sodium Acetate Addition Sequence

Table 4 shows the sequence of sodium acetate addition to the absorption.

Table 4. Effect of sodium acetate addition sequence

	Order	**Abs. sample vs. blank**	**Abs. sample vs. blank**
I	HS+SA+Fe+R+DW	0.5532	0.0806
II	HS+Fe+SA+R+DW	0.5316	0.1553
III	HS+Fe+R+SA+DW	0.5461	0.0853

HS= Hydrazin sulphate, SA= Sodium acetate, R= Reagent, Fe= Iron III Solution

Effect of Oxidizing Agent (Triple Iron Ion)

Different volumes (1-10) ml of oxidizing agent were studied. Iron ion(III) concentration of 1 × 10^{-1} M on the absorption of solutions containing different amounts of hydrazine sulfate and the results are shown in table 5.

Table 5. Effect of oxidizing agent quantity on absorption

ml of oxidant (1×10^{-1}M) solution	**Absorbance /µg of HS in 25 ml**						
	50	**100**	**250**	**500**	**1000**	**Blank**	**R^2**
1	0.1815	0.2458	0.5378	0.9435	1.7588	0.1911	0.9994
3	0.1317	0.2383	0.4721	0.6393	1.0321	0.0901	0.971
5	0.1159	0.2166	0.4382	0.6502	1.0294	0.0791	0.9753
7	0.0289	0.1162	0.1994	0.3217	0.6494	0.1010	0.9909
10	0.0499	0.0877	0.1498	0.2326	0.3426	0.0816	0.9701

Effect of the amount of reagent

The effect of different volumes (1-10) ml of the reagent at (1 × 10^{-2} M) concentration on the absorption of solutions containing different volumes of hydrazine sulfate (250µg / ml) was studied and the results are shown in Table 6.

Table 6.Effect of detector quantity

ml of reagent (1*10^{-2}M) solution	**Absorbance /µg of HS**						
	50	**100**	**250**	**500**	**100**	**Blank**	**R^2**
1	0.0099	0.0184	0.0404	0.0604	0.0956	0.0738	0.9734
3	0.0921	0.1963	0.3006	0.5008	0.8726	0.0923	0.9932
5	0.1841	0.2118	0.4009	0.6888	1.1858	0.1477	0.9988
7	0.2568	0.3562	0.6259	1.0089	1.6871	0.1556	0.9962
10	0.3216	0.4648	0.8991	1.5002	2.7654	0.2593	0.9932

Effect of iron (III) reduction time on the absorption of the product:

The effect of the time required to complete the oxidation process was studied as the solutions were left after the addition of sodium acetate solution and oxidizing agent solution to hydrazine sulfate solution for different time periods.

Table 7. Effect of tertiary iron reduction time on absorption

Time /min	**Absorbance**
Immediately	0.5563
5	0.5502
10	0.5560
20	0.5574
30	0.5572

It is noted from the results shown in the table above that the differences between the absorption of the solution at different times were few so the direct addition was adopted with little time in subsequent experiments. The effect of time was also studied after the addition of the reagent solution to the hydrazine sulfate solution and the oxidizing agent solution. The results are shown in Table 8.

Table 8. Effect of reagent addition time on absorption

Time /min	**Absorbance**
Immediately	0.5563
5	0.5539
10	0.5534
20	0.5625
30	0.5641

Effect of temperature and reaction time

The effect of different temperatures and time required to complete the reduction of Fe^{+3} to Fe^{+2} and give the highest absorption of the complex formed by using a water bath at different temperatures and for different periods of time, then the solutions were left at room temperature for 10 minutes to cool after That. The solutions were diluted with distilled water and the absorption measured for each solution versus the formal solution as shown in Table 9.

Table 9. Effect of temperature and reaction time

Temperature °C	**Absorbance / Standing time (min)**					
	10	**20**	**30**	**40**	**50**	**60**
R.T	0.0026	0.0045	0.0081	0.0101	0.0102	0.0134
30	0.0506	0.0616	0.0699	0.1568	0.1610	0.2368
40	0.1312	0.1779	0.2099	0.3119	0.3186	0.3841
50	0.2690	0.3157	0.3398	0.4125	0.4312	0.5324
60	0.3256	0.4814	0.5563	0.5591	0.5642	0.5690
70	0.4027	0.4931	0.5065	0.5241	0.5631	0.5661

Effect of superficially active substances

The effect of superficially active substances was studied by adding (3 mL) of different types of superficially active substances (positive, negative and neutral) to the reaction solution in different sequences (Table 10).

Table 10. Effect of surface active substances

Surfactant Solution 3 ml	**Absorbance / order of addition (I-V)**				
	I	**II**	**III**	**IV**	**V**
Triton X-100,1%	0.5568	0.5317	0.5349	0.5423	0.5673
C TAB,1×10^{-3}	0.5542	0.5262	0.5192	0.5235	0.5312
SDS, 1×10^{-3}	0.5549	0.5632	0.5561	0.5500	0.5553

I=HS+SA+O+R+DW,II=HS+S+SA+O+R+DW,III=HS+SA+S+O+R+DW, IV=HS+SA+O+S+R+DW, V=HS+SA+O+R+S+DW

HS=Hydrazine sulphate ,SA=Sodium acetate, O=Oxidant ,S=Surfactant ,R=Reagent

Complex Stabilization Time

For the purpose of determining the stability of the complex consisting of the reaction of iron and binary reagent ′2,2-dipride, three different amounts of hydrazine sulphate (125, 250 and 500 رامg in a final volume of 25 mL) were used. 60 ° C and left for 10 minutes at room temperature to cool and then diluted to the mark with distilled water and measured its absorption against its formal solutions at fixed intervals of 5 minutes and the results are shown in Table 11.

Table 11. Effect of time on reaction output

Time (min)	**Absorbance/µg of HS in 25ml**		
	125	**250**	**500**
0	0.3559	0.5544	0.9145
5	0.3660	0.5650	0.9312
10	0.3725	0.5881	0.9437
15	0.3848	0.6010	0.9665
20	0.3930	0.6140	0.9980
25	0.4038	0.6349	1.0135
30	0.4141	0.6486	1.0313
35	0.4177	0.6527	1.0503
40	0.4264	0.6577	1.0599
45	0.4334	0.6679	1.0706
50	0.4385	0.6735	1.0825
55	0.4463	0.6805	1.0902
60	0.4472	0.6908	1.1018

From the results shown in the table above it is noted that the product is completed after 25 minutes and remains almost stable for more than 30 minutes and this time is enough to make several measurements at the same time.

For the purpose of improving stability, a masking agents (NaF, EDTA (sodium salt)) was added at a concentration of 0.01 molar each with a volume of 3 mL and the absorbance of the model was measured according to table 12.

Table 12. Adding masking agents

Time /min	**Absorbance with EDTA 0.01M**	**Absorbance with NaF 0.01M**
0	0.3848	0.3133
5	0.4570	0.3753
10	0.5220	0.4201
15	0.5725	0.4542
20	0.6149	0.4796
25	0.6544	0.5063
30	0.6881	0.5325
35	0.7156	0.5560
40	0.7483	0.5796
45	0.7746	0.5992
50	0.7997	0.6181
60	0.8105	0.6375

Final absorption spectrum

Under the optimal conditions previously established. The final spectrum of the colored product is plotted, and Figure 1 shows the final absorption spectrum of the colored product formed versus the formal solution, the absorption spectrum of the colored product formed against the distilled water and the spectrum of the solution of the formal solution versus the distilled water.

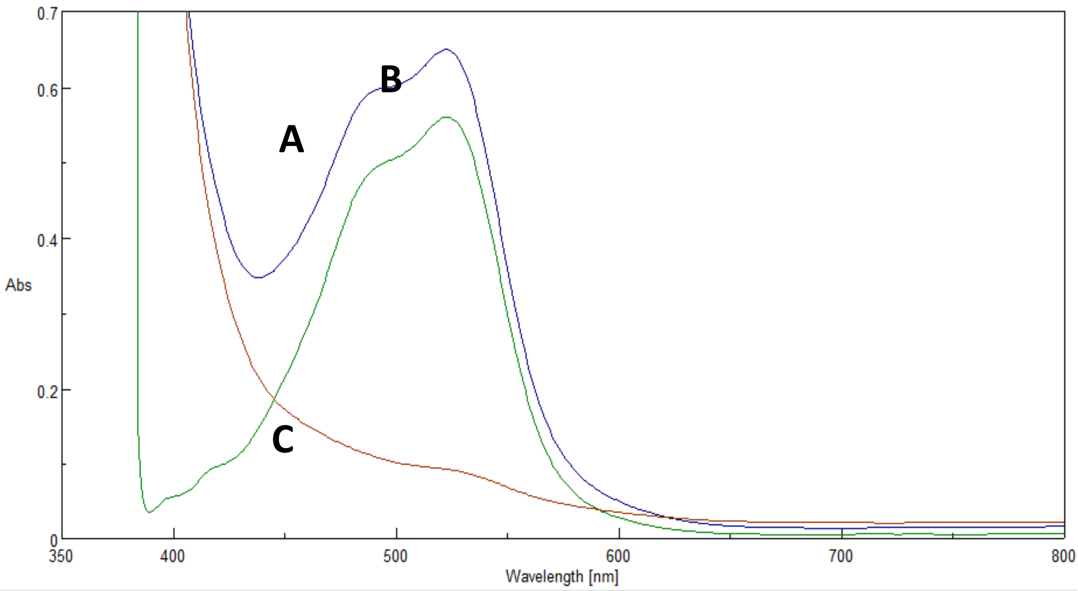


Figure 1. Absorption spectrum of 250µg /25 mL, Hydrazine sulfate is treated according to the method and measured by: (A) versus monomer, (B) versus distilled water, (C) monomer versus distilled water.

Standard working method and standard curve

The standard curve was obtained by adding increasing volumes (0.2-3.2) mL of 250µg /mL hydrazine sulphate solution to a group of 25 mL volumetric bottles and then adding 4 mL of 0.01 mL sodium acetate solution and 5 mL of oxidizing agent 1×10^{-1}M $(Fe(NH_4)(SO4)_2.12H_2O)$ and 3 mL of the reagent solution ΄2,2-bipyridyl at a concentration of 1×10^{-2}M and the solutions were placed in a water bath at 60 °C for half an hour, then Leave the bottles for 10 minutes at room temperature to cool and complete the volume with distilled water up to the mark and then measure the absorption of the complex formed at wavelength 523nm versus reagent blank as shown in Figure 2.

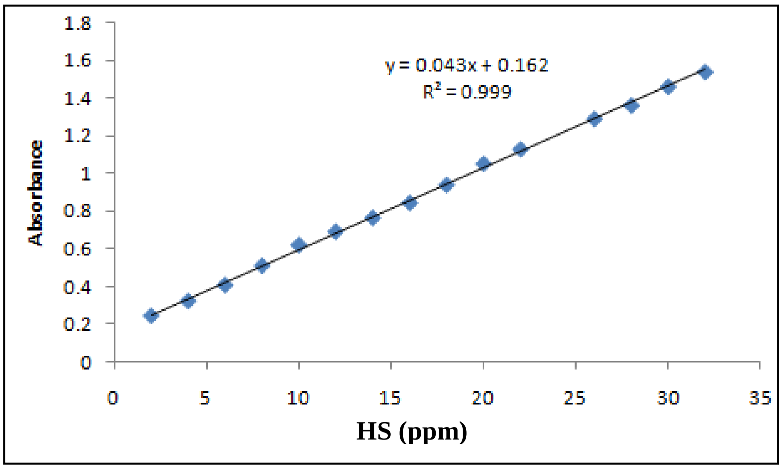


Figure 2. Standard curve for determination of hydrazine sulphate

Nature of the product formed

The Continuous variations method (Job's method) was used to find the ratio of reaction between hydrazine and ternary iron. The oxidizing agent at 1.925×10^{-3} M concentrations each was then added the optimum volumes of electrolyte solution and reagent and the solutions were placed in the water bath for half an hour at 60 °C. then, measure the absorption for each sample, the results are shown in Figure3.

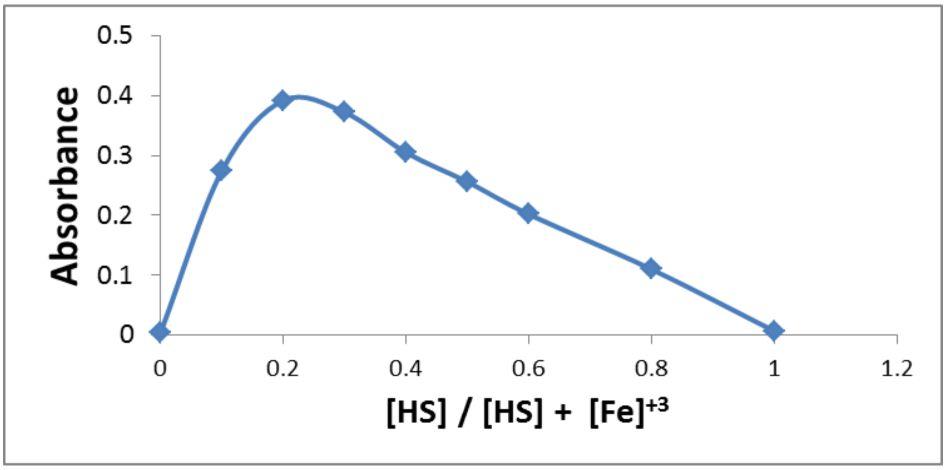
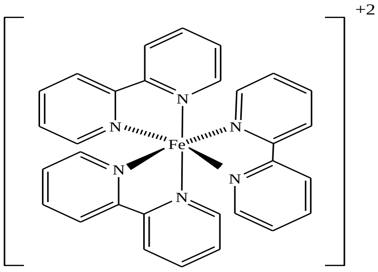


Figure **3**. Nature of the product

From Fig. 3, the molar ratio of the reaction Fe^{+3}: HS was 4: 1 and therefore the proposed reaction is consistent with the following equation

$NH_2\text{-}NH_2 + 4Fe^{+3} \longrightarrow N_2 + 4Fe^{+2} + 4H^+$

The molar ratio of the complex (Fe^{+2}-reagent) is constant and was 3: 1 and the proposed formula for the resulting complex: $[Fe\ (bipy)_3]^{+2}$



Effect of overlaps

In order to determine the method selectivity, the effect of the interference of a number of compounds was studied. These results are compared with a standard solution that does not contain any amount of interference and the results are shown in Table 13.

Table 13.Effect of interference

Foreign compound P	**Recovery % of 250 µg HS /µg foreign compound present**			
	25 µg	50 µg	100 µg	500 µg
Phenylhydrazine	123.34	139.73	153.00	275
Hydroxylamine- hydrochloride	140.2	187.8	235.12	408.89
Boric acid	99.7	102.8	105.2	107.2
Sucrose	106.9	100.5	99.9	102.2
Glucose	113.4	113.9	114.1	122.3
Urea	81.1	119.7	110.1	84.4
Thiourea	109.5	115.2	167.57	381.9
Borax	116.9	122.4	113.6	125.6
Sodium sulphate	109.4	116.7	116.3	Turbid
Sodium phosphate	133	107.5	109.9	Turbid
Sodiun persulphate	105.1	102.2	95	3.4
Glycerol	123.8	122.8	121	112.6
Sodium nitrite	16.7	40.8	36.4	106.6
Sodium nitrate	122.4	90.8	115.4	108.9
Sodium chloride	84.5	93.8	83	87.1
Potassium perchlorate	108.3	92.4	101.8	95.2
Ammonium chloride	113	103	102.3	110.9

Application Method

The proposed method for estimating hydrazine was applied in different water models such as tap water, river water (Tigris River, Rashidiya area) and sea water (Mediterranean). The results are shown in the table below:

Table 14. Results of application of method on water types

Sample	ml of sample	Recovery , (%)
Tap water	1	98.5
	3	95.35
	5	95.8
	10	96.88
River water	1	97.5
	3	96.8
	5	95.1
	10	95.3
Sea water	1	97.5
	3	90.5
	5	91.6
	10	91.7

Comparison of the two methods used to estimate hydrazine

A comparison has been made between the present methods with the literature[12] used to estimate hydrazine. From the table below, the present method have a good analytical properties and can be applied successfully in aqueous models.

Table 1**5**. Comparison of the method

Analytical parameter	Present method	literature method[26]
pH	4.29	2.9
Temperature ,C°	R.T	60 C°
λ_{max} nm	730	523
Reagents used	3 reagents	3 reagents
Principle reagent	Potassium ferricyanide	2,2'-bipyridyl
Time of analysis , min	30 min	30 min
Determination range	1-22 ppm	2-32 ppm
Molar absorptivity	1.95×103	5.6×10^3
Redox stoichiomstry	1:4	1:4
Color	Prussian blue	Red-pink
Recovery%	100.1	100.8
RDS%	$\pm$0.2	$\pm$1.25
Determination coefficient (R^2)	0.990	0.999
Application	Could not be applied determination of hydrazine in waters	Has been applied successfully for determination of hydrazine in sea, river and tap water

Conclusions

A rapid and sensitive spectroscopic method for estimating hydrazine was developed. The method is based on the reduction of hydrazine triple iron ion to the binary iron ion which in turn reacts with 2,2-di-pyridil reagent to produce a complex (red-pink) color that is soluble and stable for a period of 60 minutes. At least it has a maximum absorption at a wavelength of 523 nm. The standard straight curve shows the PIR law following limits of concentrations (50-800) µg / 25 mL (2-32) ppm, and the standard relative deviation values (RSD) were Depending on the concentration level, the molar absorption coefficient was 103 × 5.6 liters.

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