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Spectroscopy Determination of Iron (III) Ion by Using Azo Dye Derived from Mefeminamic Acid

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Abstract

By coupling diazonium salt of 5-mercapto-4H-1,2,4-triazol-4-yl)diazenyl with 3-nitrophenol, triazole azo dye (Y2) was obtained. They represented good complexing properties towards Fe(III) ions, resulting in colored complexe. In this paper, the use of the synthesized triazole azodyes as reagents in spectrophotometric determination of Fe(III) was presented. Absorption maxima, optimum pH values, molar extinction coefficients and stoichiometric ratios were determined. Y2 was found to be the best chromogenic reagent due to its high selectivity and sensitivity towards Fe(III) ion. Y2 was successfully applied with high precession and good accuracy to the spectrophotometric determination of Fe(III) ion in solutions.

Keywords: Mefenamic acid, Azo dye, Iron(III), Spectroscopy.

Introduction

Mefenamic acid, with the chemical formula $C_{15}H_{15}NO_2$, is also referred to as 2-[(2,3dimethylphenyl) amino] benzoic acid (Scheme 1). It has a molar mass of 241.28 $g.mol^{-1}$ and appears as a white to off-white, microcrystalline powder. Its melting point is (230-231)ºC. Mefenamic Acid is prescribed for the management of mild discomfort and dysmenorrhea **[1]**.

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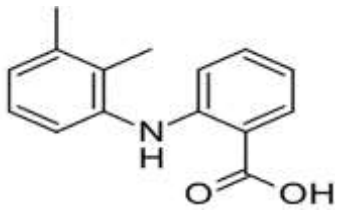

Scheme 1: The mefenamic acid structure [1] .

Mefenamic acid has been extensively referenced in scientific and medical literature, and the mefenates are among the most thoroughly researched pharmacological drugs. Shortly after its discovery in 1963, mefenamic acid quickly became widely accessible to the public and was frequently recommended as a pharmaceutical. During the 1990s, certain rankings placed it inside the top three nonsteroidal anti-inflammatory drugs (NSAIDs) that were commonly recommended **[2]**. It is widely utilized in the management of many conditions such as rheumatoid arthritis, osteoarthritis, non-articular rheumatism, and sports injuries.

Fortifying food with iron is a strategy used to provide our bodies with this essential metal. The choice of supplements is crucial because of its potential for effective absorption. Ferritin is a metalloprotein that may accumulate iron and is easily absorbed by the body. Iron buildup takes place when there are large levels of iron salts in the plant's growing environment. Over-expressing ferritin has become a technique for adding iron to food goods **[3]**. In recent years, the literature has documented numerous novel approaches to quantify the concentration of iron in solutions, food, and other substances. Colorimetric methods have been employed in speciation analyses for iron forms with concentrations over 1mg/kg **[4]**.

Accurate and expedient quantification of iron concentration in aqueous solutions holds significant importance for numerous sectors. Manufacturing firms that require the cleaning of metal parts may need to assess the concentration of iron in their waste streams to ensure compliance with environmental regulations. Government entities at various levels have a vested interest in conducting tests on wastewater, natural waters, and drinking waters to ascertain the levels of iron present. This is done to ensure adherence to legal requirements and to guarantee the safety of the water supply for both animals and the human population. Advanced laboratories with contemporary equipment can analyze the iron concentration using atomic emission spectroscopy in an inductively coupled plasma (ICP) spectrometer. Flame atomic absorption

spectrometry can also be utilized, however, it is important to note that iron solutions have a tendency to obstruct the burner with iron oxide when the attention surpasses a specific threshold **[5,6]**. Both of these methods necessitate a substantial allocation of resources towards acquiring specialized equipment and establishing a well-maintained laboratory setup that includes handling pressurized gases and managing exhaust fumes. The solution chemistry involved in a colorimetric assessment of iron concentration is quite uncomplicated and may be simplified into a kit format. This allows for the analysis to be conducted either in the field using portable equipment or in a laboratory using an affordable visible spectrophotometer and basic glassware. This work presents a technique for identifying minute quantities of iron in various sample types, including pure substances, by utilizing a reagent generated in a laboratory setting. Upon investigation, it was determined that the product had a distinct coloration when it reacted with iron, resulting in the formation of a complex with the maximum absorption at a wavelength of 545nm. The procedure proved to be efficacious and precise in measuring the iron content in the utilized samples.

Experimental

Apparatus: The infrared spectra of the produced compounds were recorded on a potassium bromide (KBr) disk and evaluated using Fourier Transform Infrared Spectroscopy (FTIR). Spectrophotometer PERKIN ELMER SPEACTRUM-65/ Germany. The JASCOV-650 is a twin beam UV-Vis spectrophotometer from Japan, fitted with a 1cm quartz cell. It is also capable of doing ^{1}HNMR analysis. The spectra were acquired using a Bruker 400 MHz spectrometer at the Central Laboratory.

Materials and Methods

We obtained the analytical reagent grade chemicals from BDH and Panreac. The State Company for Drug Industries and Medical Appliances (SDI), located in Samara-Iraq, generously supplied the standard powder of mefenamic acid. Tap water sample (drinking water system in June, Baquba, Diyala), river water (Diyala River, Baquba, Diyala), and industrial wastewater from the Diyala Electrical Industry Company (industrial wastewater from Electrical manufacturing factory), juice bottles in the local markets in Baquba.

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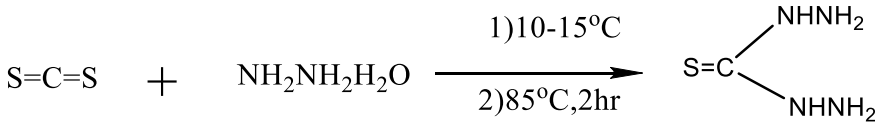
Synthetic methods of the compounds:

Synthesis of Thiocarbohydrazide [y1]

Hydrazine hydrate (25.03 g, 0.5 mol) was placed in a Beaker and placed in an ice bath with a temperature of 10 ° C and then added (7.61 g, 0.1 mol) of carbon sulfide drip into the flask while keeping the temperature below 15 ° C for 15 minutes with continuous stirring and after completion of the addition leave the solution for 20 minutes then reaction mixture was refluxed for 4 hours at 85 °C . Scheme (2) show the general reaction to synthesis of thiocarbohydrazide [Y1].The completion of the reaction was checked by TLC (mobile phase Hexane: Ethyl acetate) Where it was noted that the reactions completed and it was pure product. The reaction mixture was subjected to cooling, followed by filtration of the resulting precipitate, which was subsequently washed with water **[7]**. The physical properties of the synthesized compound (Y1) is given in (Table 1).



Scheme 2: synthesis of thiocarbohydrazide [Y1]

Synthesis of 4-amino-5-(2-((2,3-dimethylphenyl)amino)phenyl)-4H-1,2,4-triazole-3-thiol [Y2]

(2.4 g,0.01 mol) of mefenamic acid was mixed with thiocarbohydrazide [Y1] (1.59 g, 0.015 mol) and then transferred to the melting tube. The mixture was placed in a oil bath at a temperature of (200-202)°C for 3 hours, then 10% sodium bicarbonate was added to the precipitate formed in the melting tube for 2 hours, then filtered and washed the precipitate well with distilled water **[7,8]** . The purity of the chemical was verified by TLC (using a mobile phase of Hexane: Ethyl acetate). The title compounds were obtained by recrystallizing the result from ethanol. The Table (1) provides the physical properties of the produced chemicals (Y2). The proposed mechanism for the synthesis of chemical Y2 is presented in scheme (3).

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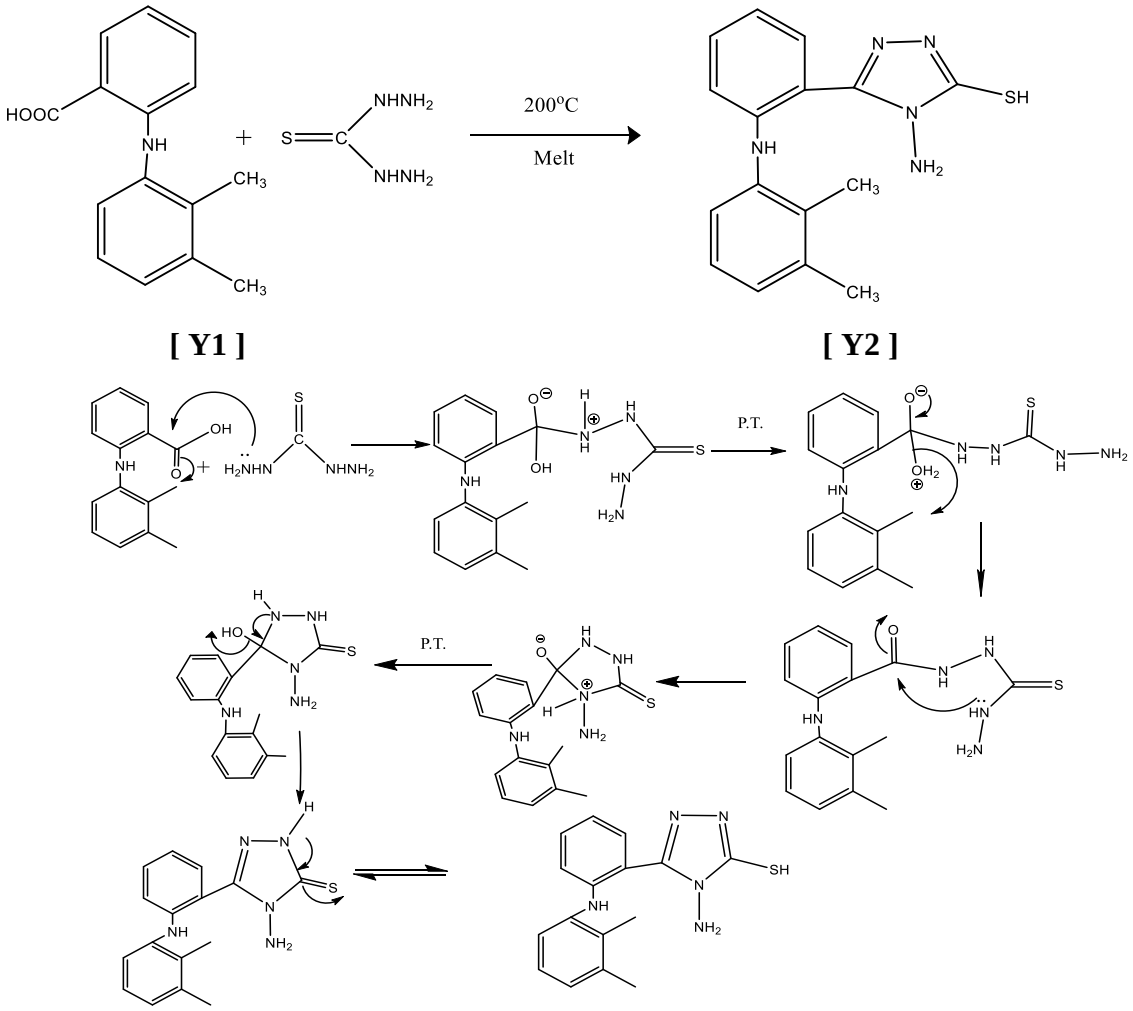

Scheme 3: The general mechanism to synthesis of 4-amino-5-(2-((2,3-dimethylphenyl)amino)phenyl)-4H-1,2,4-triazole-3-thiol [Y2]

Synthesis of compounds [Y3]

Take 0.001 moles of 4-amino-5-(2-((2,3-dimethylphenyl)amino)phenyl)-4H-1,2,4-triazole-3-thiol [Y2] and dissolve in mixture of (10 ml H_2SO_4, 10 ml ethanol and 10 ml distilled water) and place in an ice bath (0-5)^oC. Then dissolved 0.0011 moles of $NaNO_2$ in 5 ml of distilled water and place in ice. Then add $NaNO_2$ to the first solution gradually and left in the ice bath for a quarter of an hour. Dissolve 3 g of NaOH in 27 ml of distilled water and add 3-nitrophenol with a number of moles of 0.001 mol and placed in an ice bath and then add this solution to the first prepared solution gradually while maintaining the temperature of the ice bath (0-5)^oC and

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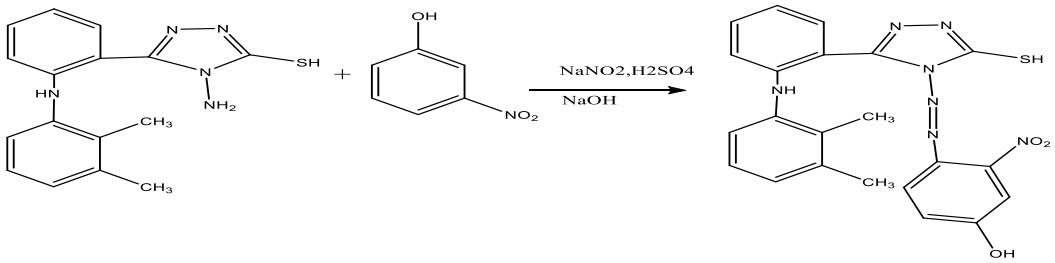
continue stirring for half an hour or more and then leave it to filter **[9]** . Scheme (4) show the general reaction to synthesis of [Y3].



Scheme 4: synthesis of compounds [Y3]

Table 1: Physical properties of synthetic substances

Comp. No.	Yield %	Appearance	M. P. °C	Recrystallization solvent	Retention Factors
Y1	88	White	180-182	Ethanol	0.34
Y2	85	Brown	245-248	distilled water	0.41
Y3	60	Dark red	148-150	Ethanol	0.29

Reagent working solution (0.0026 M)

A total of 0.3110 grams of the compound 4-((3-(2-((2,3-dimethylphenyl)amino)phenyl)-5-mercapto-4H-1,2,4-triazol-4-yl)diazenyl)-3-nitrophenol was completely dissolved in 25 milliliters of pure water.

Solutions of Cation

Cation solution with a molecular formula ($Fe(NO_3)_3.9H_2O$) at a concentration (1000ppm) was prepared by dissolving the calculated weight (0.7234gm) in 100mL distilled water.

Determination of λmax of Fe (III) complex

The primary test involved combining 1 mL of a standard Fe (III) ion solution with a concentration of 200 ppm, with 1.0 mL of a compound called 4-((3-(2-((2,3-dimethylphenyl)amino)phenyl)-5-mercapto-4H-1,2,4-triazol-4-yl)diazenyl)-3-nitrophenol (Y3) at a concentration of 0.0026 M. After adding the ingredients to a 10 mL volumetric flask, the mixture was shaken gently until a deep crimson hue emerged. The flask was then filled up to the mark with distilled water and the resulting solution was scanned across the entire UV-

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VIS range of (200 - 1100) nm, using a blank as a reference. The resulting spectra revealed a λmax of 545 nm.

General recommended procedure for calibration

1.0 mL portions of a Fe(III) standard solution with a concentration ranging from 5 to 700 parts per million (ppm) were put into a set of 10 mL volumetric flasks. Each flask was supplemented with 2 mL of a reagent solution with a concentration of 0.0026 M. After a duration of 10 minutes, to get to the specified mark, the contents were diluted with distilled water. After that, they were given five minutes to themselves. Subsequently, the absorbance was measured at a wavelength of 545 nm in comparison to the reagent blank.

Results and Discussion

The structure of compound (Y2) were confirmed by FTIR and ^{1}HNMR spectroscopy. FTIR figure (1) show the appearance of stretching vibration (asymmetric and symmetric) for (NH_2) group at (3366, 3322) cm^{-1}, band at (3133) cm^{-1} for (NH). Absorption band at (3087) cm^{-1} was due to (C-H) aromatic. (2935, 2917) cm^{-1} was assigned to and aliphatic (C-H), appearance of new clear absorption band at (1635) due to (C=N) of triazole ring. The stretching frequency at (1578 and 1494) cm^{-1}, which corresponds to the (C=C) group, was observed. Additionally, the elimination of one carbonyl stretching vibration band confirmed the creation of this compound.

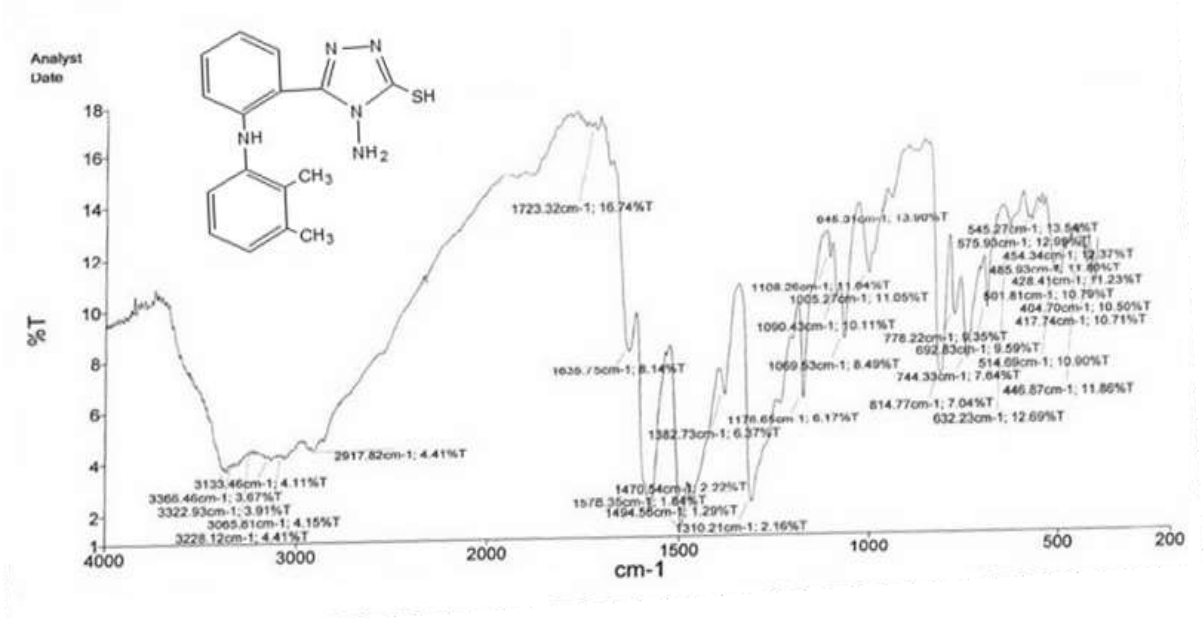

Figure 1: FTIR spectrum of compound (Y2)

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The [1]HNMR spectrum of compound (Y2) in δ ppm, Figure (2) indicates a singlet signals at (12.30) due to (1H, SH), while singlet signal at (12.08) ppm for (NH). There are seven aromatic protons that have been ascribed to several signals ranging from 8.26 to 6.70. The singlet signal at 5.76 ppm is caused by the presence of 2 hydrogen atoms and an amino group (NH_2). Additionally, the signal at 2.13 ppm is due to the presence of 6 hydrogen atoms and two methyl groups (2xCH_3).

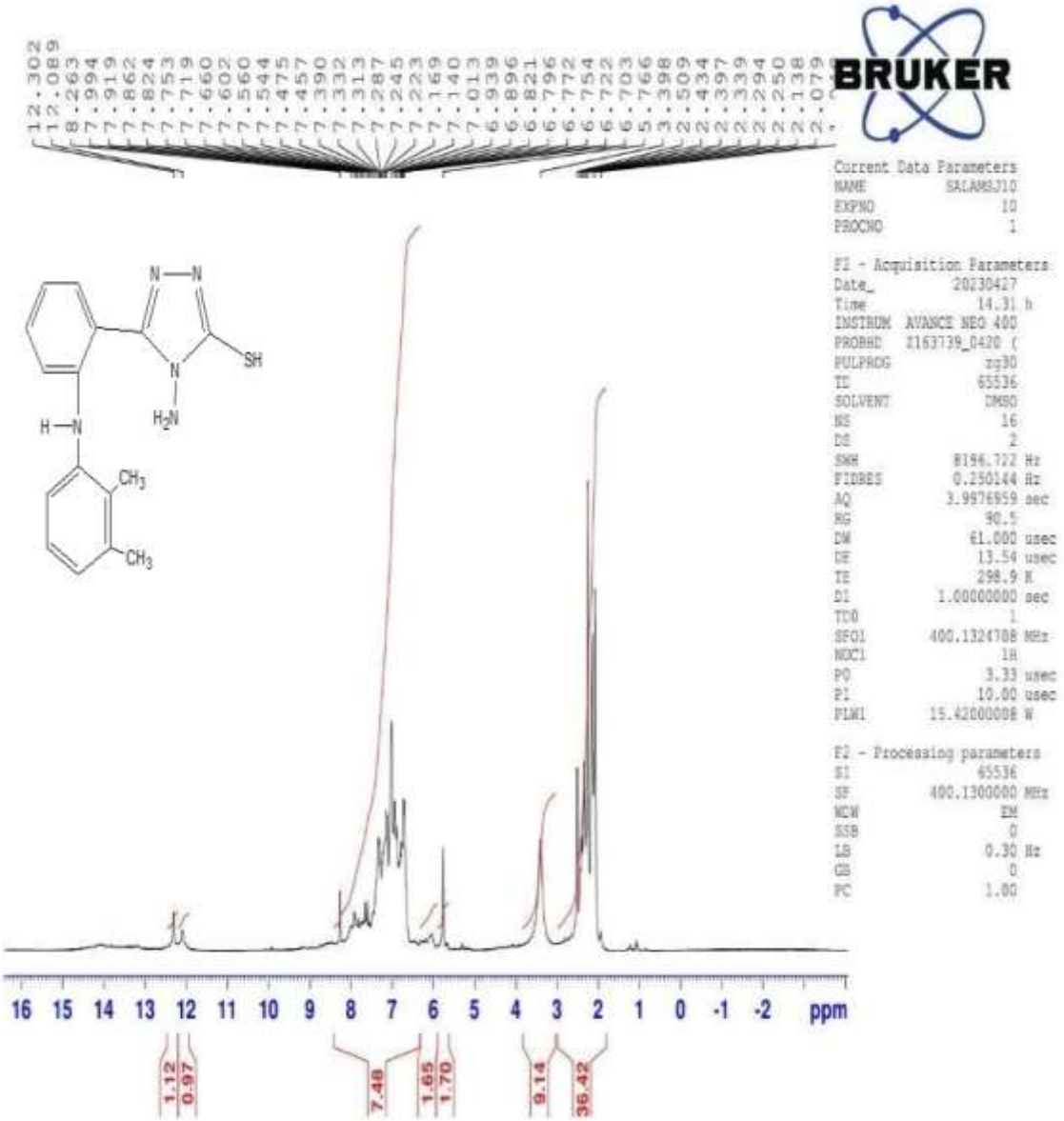

Figure 2: [1]HNMR spectrum in of compound (Y2)

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The structure of chemical (Y3) was verified using infrared spectra and ^{1}HNMR spectroscopy. The FTIR figure (3) displays a peak at (3425) cm^{-1} corresponding to the (NH) group, and the absorption band of OH overlaps with NH. The presence of an absorption band at 3078cm^{-1} can be attributed to the (C-H) aromatic group.The absorption band at (2921,2842) cm^{-1} was assigned to an aliphatic (C-H) bond. The absorption band at (1625) cm^{-1} was attributed to the (C=N) bond of the triazole ring. The appearance of an absorption band at (1594) cm^{-1} was a strong indication of the presence of the (N=N) bond in the azo compound, confirming the creation of this compound. The stretching frequency at (1574 and 1491) cm^{-1}corresponds to the (C=C) group, whereas the other bands at (1527 and 1318) cm^{-1} are attributed to the stretching vibration of (NO_2).

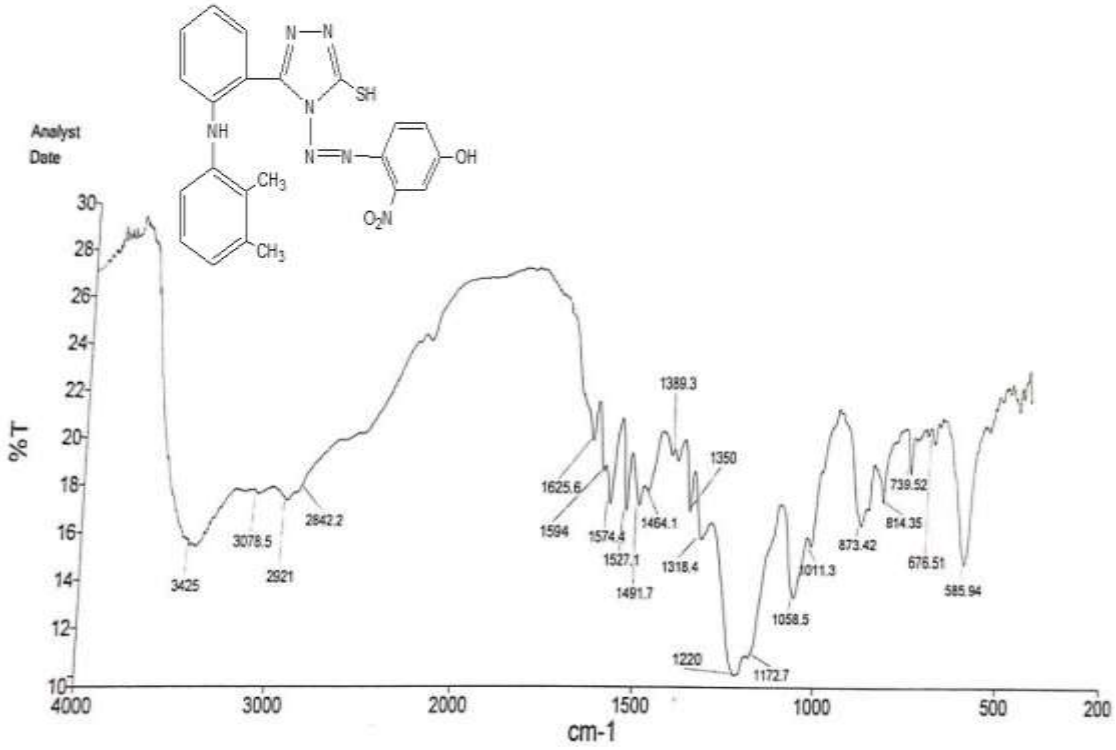

Figure 3: FTIR spectrum of compound (Y3)

Figure (4), ^{1}HNMR spectrum of compound (**Y3**) in δ ppm: shows a singlet signal at (9.63) ppm for (1H,SH), singlet signal at (8.29) ppm due to (1H,NH) Multiplet signals between (7.22-6.60) assigned to ten protons of aromatic ring, whileas singlet signal at (5.13) due to(1H,OH), and the signal at (2.20-2.16) ppm due to (6H,2xCH_3).

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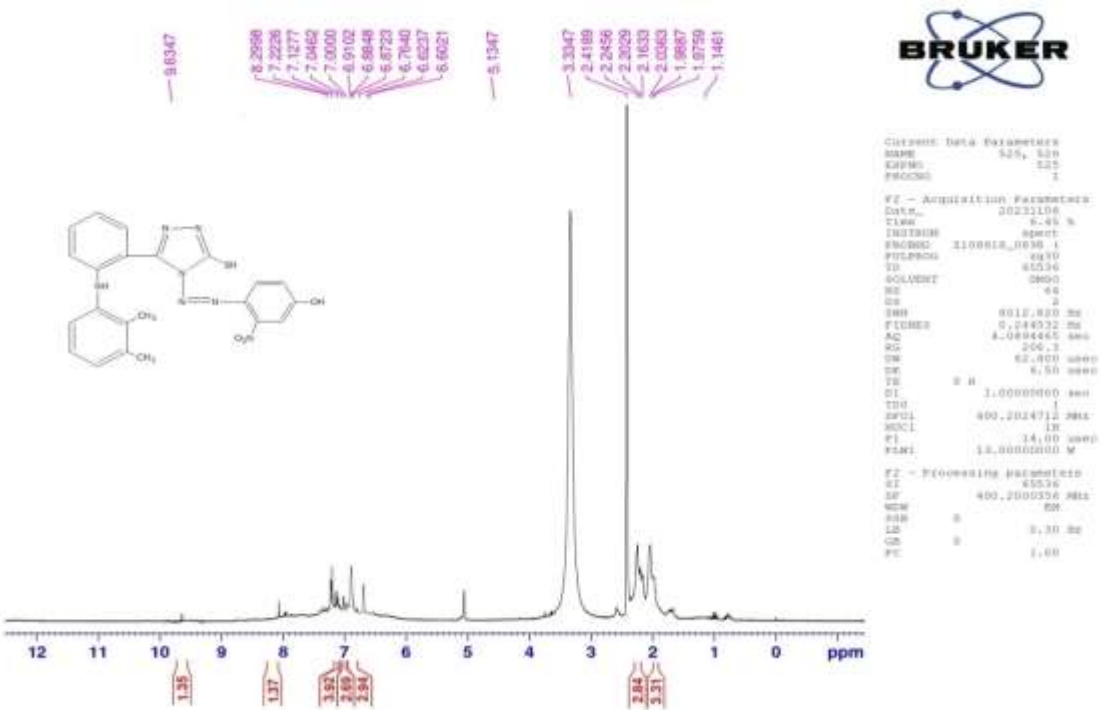

Figure 4: [1]HNMR spectrum in of compound (Y3)

Spectrophotometric Determination of Fe (III) ion with the reagent [Y3]

Studies Absorption spectra at primary test

The UV- visible spectrum of the synthesized reagent (0.0026 M) (Y_3) after dissolving in 0.01M H_2SO_4, the spectrum shows two main maximum absorption peaks Figure (5). The first at wavelength of λmax at 239 nm is due to transitions (π- π*) in imidiazole ring **[10]** and the second peak at λmax of 280 nm due to the transition of (π-π*) type in the benzene ring **[11]** .

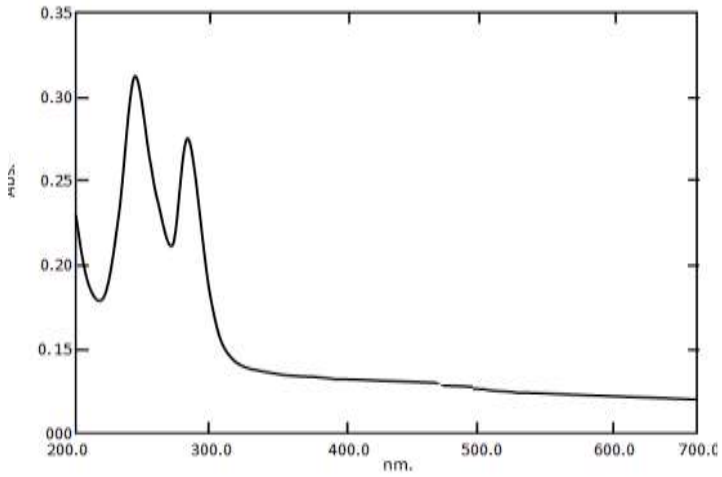

Figure 5: UV-Vis. spectrum of organic reagent (Y3)

To limited λmax for complex of Fe(III) with(Y3), 1mL aqueous solution contain 20 µg of Fe(III) then add 1mL of (0.0026 M) (Y3) and mixed well , the spectrum was as in Figure (6) shows λmax at 545nm is the wave length for maximum absorbance of complex against reagent blank .

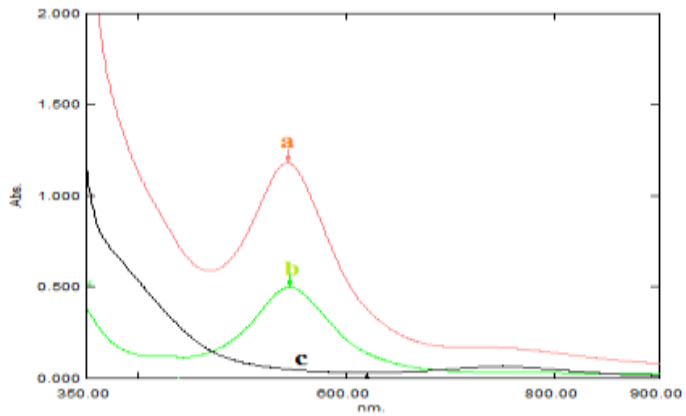

Figure 6: Absorption spectra of: **(a)** 20 µg.mL^{-1} Fe (III) against reagent blank under optimum conditions , **(b)** 20 µg.mL^{-1} Fe (III) against reagent blank under primary test conditions, **(c)** blank solution against distilled water.

Optimization of reaction variables

A comprehensive investigation was conducted to analyze the impact of different parameters on the formation of color products. This was achieved by systematically altering one parameter at

a time while keeping all other parameters constant. The variables encompass the reagent volume (Y3), as well as the reaction time, temperature, and the stability of the resulting product.

1. Effect of the volume of reagent solution

An investigation was conducted to examine the impact of reagent volume on the formation of the [Fe (III)] complex with the reagent [Y3]. The findings of this study are presented in Figure 7.

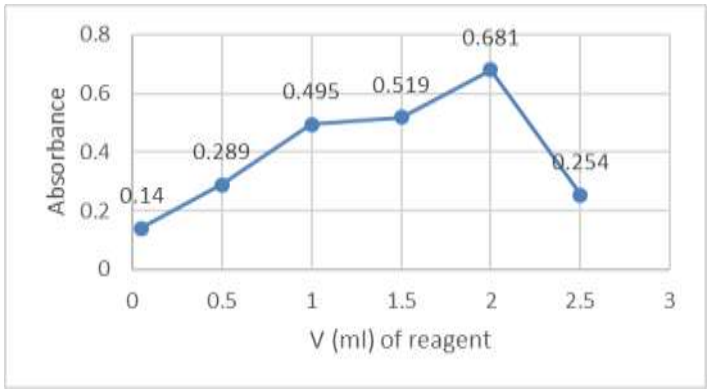

Figure 7: The effect of the volume of the reagent on the absorption

It was found that 2.0 mL of (0.0026) M (Y3) was enough to develop the absorbance to its maximum intensity, beyond which the absorbance slightly decreased, while the absorbance intensity did not affect at lower volume of the π-acceptor reagent.

2. Effect of coupling reaction time

The study investigated the impact of reaction time on the formation of the colored complex, while a constant concentration of Fe (III) at 20 $\mu g.mL^{-1}$ was reacted with reagent (Y3). Measurements of absorbance were taken at various time intervals .starting from right away until a minimum of twenty-five minutes. The data indicates that the reaction reached completion within a time frame of 10 minutes, as presented in Table (2).

Table 2: Time of reaction affects absorption

reaction time	Abc
5 min	0.681
10 min	**0.904**
15 min	0.873
20 min	0.640
25 min	0.418

Effect of temperature

Temperature impacts on the formation complex were examined in this study. Table (3) shows how temperature affects this compound. The given results show that the absorbance attains a maximum colour at temperature 25 ºC. The absorption values then decrease with increasing temperature the reason may be due to a decrease in stability or as a result of its disintegration at high temperatures **[12]** .

Table 3: The effect of the temperature of reaction on the absorption

T	Abc
10°C	0.595
25°C	0.904
35°C	0.751
50°C	0.286

The stability

By letting the reaction run for varying amounts of time, we were able to study how time affected the final result. The recommended process determined that 5 minutes is the optimal time. The color that was created stayed stable for 60 minutes at room temperature. This stability period is long enough to allow several sequential measurements to be performed. Please refer to Figure 8 for more details.

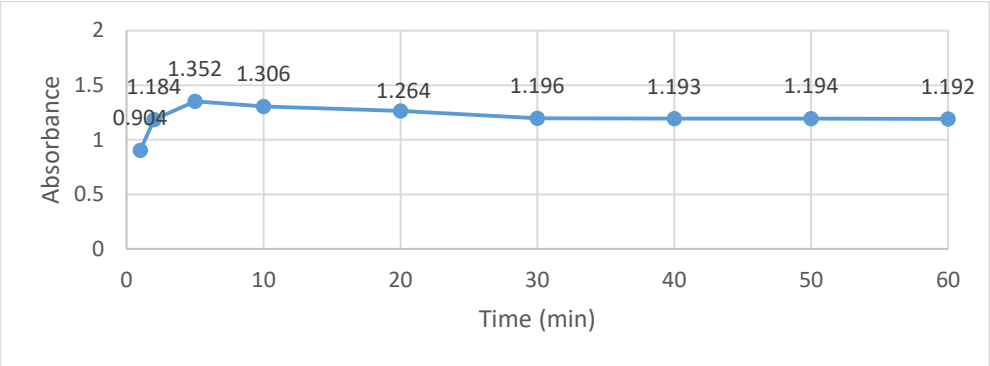

Figure 8: Time affects stability

Calibration curve of Fe(III):

A calibration curve is constructed to quantify the amount of Fe(III) in an aqueous solution, using the proposed method. This procedure involves utilizing a 1mL aqueous solution containing a range of Fe(III) ions ranging from 5.0 to 700.0 µg. Subsequently, 2mL of reagent (Y3) is added, and the absorbance is measured at the wavelength of maximum absorption (λmax) of 545nm. The results corresponded to those depicted in Figure (9).

The limit of detection (LOD) refers to the minimum concentration of an analyte that can be detected reliably, although not necessarily quantified. On the other hand, the limit of quantitation (LOQ) represents the lowest concentration that can be determined with acceptable accuracy and precision**[13]**. These values were calculated using the following expressions: (3.3 × SD)/ S for LOD and (10 ×SD)/ S for LOQ. Here, "SD" denotes the standard deviation of the blank**[14]**, and "S" represents the slope of the calibration line.

The regression equation, correlation coefficient, molar absorptivity (how effectively a substance can absorb light of appropriate wavelength) **[15]**, Sandell's sensitivity **[16]**, LOD and LOQ are calculated and listed in Table (4).

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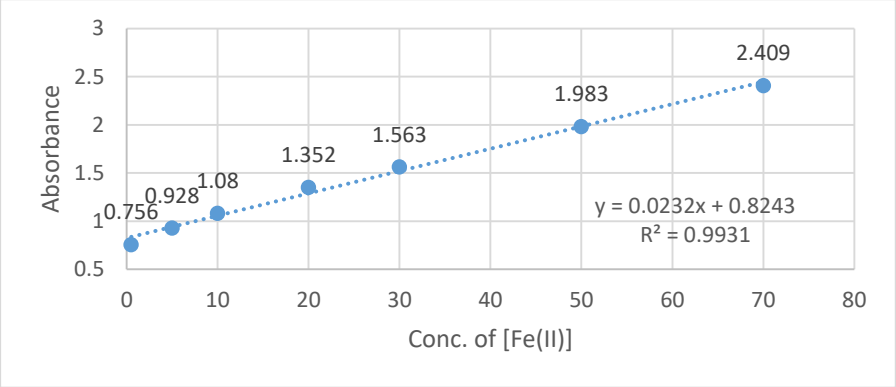

Figure 9: Calibration curve for Fe(III) ion

Table 4: Optical characteristics, statistical data of the regression equations and validation parameters of Fe(III)

Parameter		Value
Optical characteristics		
1.	λ_{max} (nm)	545
2.	Apparent molar absorptivity ($L.mol^{-1}.cm^{-1}$)	23803.2
3.	Sandell's sensitivity ($\mu g.cm^{-2}$)	0.0431
Regression analysis		
1.	Slope (mL. μg^{-1})	0.0232
2.	Intercept	0.8243
3.	Regression coefficient (r)	0.9931
Validation parameters		
1.	Beer's Law Limit (Linearity, $\mu g.\ mL^{-1}$)	0.5-70.0
2.	Limit of detection ($\mu g.\ mL^{-1}$)	0.0562
3.	Limit of quantitation ($\mu g.\ mL^{-1}$)	0.1607

Stoichiometric Ratio Determination

The Job's continuous variation approaches **[17]** and molar ratio **[18]** are utilized to ascertain the optimal ratio between the Fe(III) medication and Y3 reagent. The figures (10 and 11) demonstrate that the interaction between Fe(III) ions and the reagent takes place on an equimolar basis. The two straight lines connect at a ratio of 1:2 [Fe(III)]. [*Y3*].

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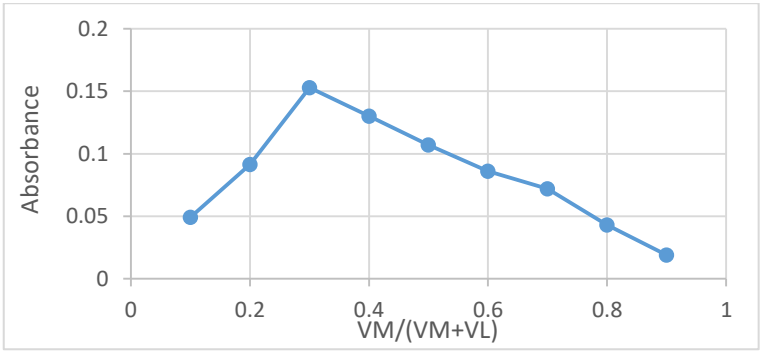


Figure 10: Fe(III)-Y3 reaction continuous variation method

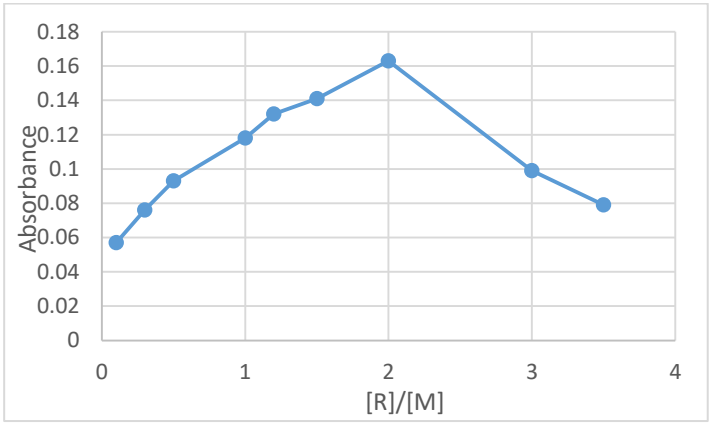
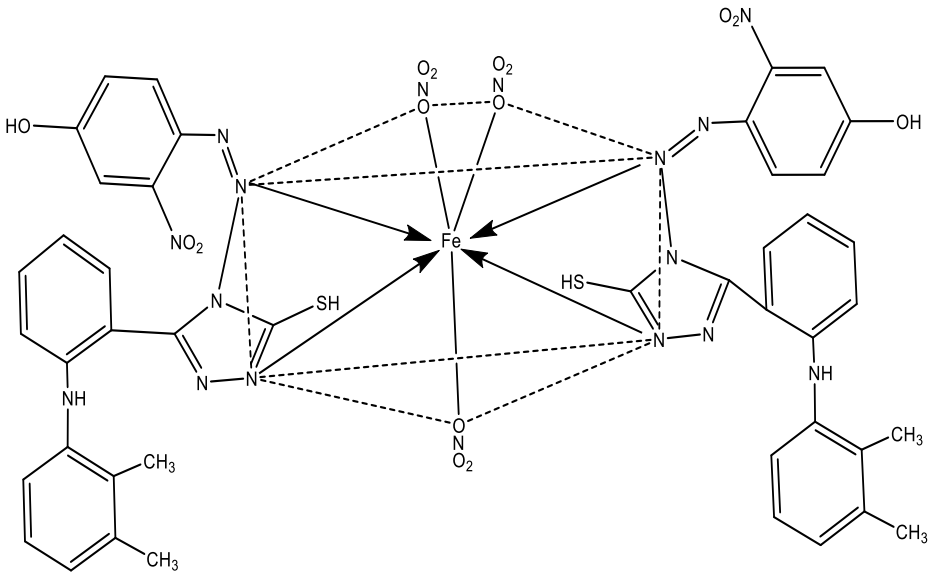


Figure 11: Mole ratio method for reaction Fe(III) with Y3



Scheme 5: The proposed form of the resulting complex

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The effect of interference and masking agents

Interference effect

To estimate the effect of the presence of cations as interferers when estimating the iron ion, a number of cations were chosen to determine the extent of their influence on the absorption of the iron ion, as shown in Table (5).

A number of anions were also chosen to study their effect on iron ion estimation. The results of the study appear in Table (6)**.**

Table 5: Evaluation of cations interference for the determination of Fe (III) by propose method

Cations	Chemical formula	25 µg/mL		50 µg/mL	
		Valuable Absorption after adding cations	Relative error E%	Valuable Absorption after adding cations	Relative error E%
-	-	1.352	-	1.352	-
Cu(II)	$Cu(NO_3)_2.4H_2O$	0.659	51.257	0.745	44.896
Ag(I)	$AgNO_3$	0.821	39.275	0.841	37.795
Pb(II)	$Pb(NO_3)_2$	0.745	44.896	0.507	62.500
Mg(II)	$Mg(NO_3)_2.4H_2O$	0.607	55.103	0.935	30.843

$RE\% = ((A_{true} - A_{cal})/A_{true})100$

Table 6: Evaluation of anions interference for Fe (III) determination by proposed method

Antions	Chemical formula	25 µg/mL		50 µg/mL	
		Valuable Absorption after adding anions	Relative error E%	Valuable Absorption after adding anions	Relative error E%
-	-	1.352	-	1.352	-
$SO_4^{=}$	K_2SO_4	0.305	33.062	0.856	36.686
Br^-	KBr	0.252	44.378	0.912	32.544
SCN^-	KSCN	1.092	19.230	1.197	11.464
CN^-	KCN	0.981	27.440	0.904	33.136

Masking agent

A method was proposed to remove the effect of interference, and based on that, four masking agents were chosen to determine the extent of the effect of the competition process between them and the reagent on the reaction, 1 ml of a masking agent was added to remove the ions causing the interference and block them, table (7) shows the results.

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Table 7: Effect of masking agents

NO.	MasKing agent(0.1M)	Abs. of 20 µg/mL Fe (III)
1	Without MasKing agent	1.352
2	Thiourea	0.591
3	Ascorbic acid	0.704
4	Na2 EDTA	1.004
5	KCl	1.355

The results in Table (7) show that potassium chloride can be used as a masking agent when measuring iron ions better than other agents that cause the complex to dissociate.

Application

In clean polyethylene bottles, samples were taken of the following: tap water sample (drinking water system in June, Baquba, Diyala), river water (Diyala River, Baquba, Diyala), and industrial wastewater from the Diyala Electrical Industry Company (industrial wastewater from Electrical manufacturing factory), juice bottles in the local markets in Baquba. With the exception of tap water, the substances were passed through filter paper and then kept in a refrigerator at a temperature of 4 °C. Finally, 100 ml of each sample was used to measure the amount of studied ions present in these samples by following the proposed method.

1ml and 2 ml are taken from each sample, and the method of work is applied to it, and the absorption is measured for Fe (III) ion separately at λ_{max} . With optimal conditions were used to form the Fe (III) complex .The process is repeated three times. The results are shown in the tables (8) below

Table 8: Measurement of absorbance of ion Fe (III) in samples

Determination of Fe(III) ion				
Samples	Sample volume	Added (µg/mL^{-1})	Found*(µg/mL $^{-1}$)	Recovery (%)
Tap drinking water	1ml	-	0.003	-
		10	10.007	100.07
	2ml	-	0.005	-
		10	10.009	100.09
Diyala River	1ml	-	0.101	-
		10	10.134	101.340
	2ml	-	0.153	-
		10	10.240	102.400
industrial wastewater	1ml	-	0.404	-
		10	11.048	110.480
	2ml	-	0.669	-

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		10	11.113	111.130
juice	1ml	-	0.002	-
		10	10.001	100.010
	2ml	-	0.003	-
		10	10.002	100.010

Measuring three replicate Recovery= (x cal/x)100

To assess the suitability of the suggested technique for detecting Fe(III) ions in water samples.The Fe(III) ion quantification method was used to analyze tap water samples from the drinking water system in June, river water from Diyala River, industrial wastewater from the Diyala Electrical Industry Company, and bottles of juice from local markets in Baquba, Iraq. The samples were collected in Baquba, Diyala, Iraq, and stored in the refrigerator without any pretreatment until analysis. In order to verify the presence of the examined ion and measure its concentration in different genuine water samples, the analysis was conducted without adding any other substances. Based on the data presented in Table 8, the investigated ion was not detected in tap water and juice samples. However, a substantial amount of the ion was found in Diyala river water and the industrial effluent from the company. The standard ionic solution of the examined ion was added to the genuine samples at a concentration of 10 mg/mL, using the appropriate volume of samples. The obtained relative recovery values (RR%) ranged from 100.010 to 111.130%, demonstrating the suitability and precision of the suggested approach for quantifying the specified ion in the real samples tested, without any notable matrix influence.

Conclusion

A simplified and expedited spectrophotometric method for detecting iron ions was developed using a reagent called [Y3]. The reagent was prepared using the traditional method of dualization of diazonium salts of a compound called 4-amino-5-(2-((2,3-dimethylphenyl) amino) phenyl) -4H-1,2,4-triazole-3-thiol of the derivative (3-nitro phenol). This method proved to be more straightforward and efficient. The parameters facilitated the swift and accurate measurement of iron ions in four samples. The suggested method did not necessitate temperature regulation.

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