EXTRACTIVE SPECTROPHOTOMETRIC DETERMINATION OF MICROAMOUNTS OF Fe(III) IN WATER SAMPLES USING A NOVEL PYRAZOLIDINYL-BASED REAGENT

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Received 18.09.2025
Accepted 17.12.2025

Abstract. *This article presents the study of the complexation behavior of (E)-3-(((1-methyl-3-oxo-2-phenyl-5λ³-pyrazolidin-4-yl)diazenyl)methyl)pentane-2,4-dione (R_1) with Fe(III) ions using the extractive spectrophotometric method. Different organic solvents were evaluated as extractants, among which 1-butanol provided the highest extraction efficiency. Optimal conditions for complex formation and extraction were established, revealing that Fe(III) predominantly exists as $Fe(OH)_2^+$ under the experimental conditions at pH=5. The absorption spectrum of the Fe(III)-R_1 complex was recorded across a pH range of 1–6, showing maximum absorbance at pH 5 and λ_{max} = 540 nm. The method obeyed Beer's law over the concentration range of 0.112–3.36 μg/mL. The Sandell sensitivity was calculated as 0.0049 mg/cm², and the extraction efficiency reached 99.4%. The detection limit was determined to be 0.032 μg/mL. The method was successfully applied to the determination of Fe(III) in environmental water samples from the Zira district of Baku. Results were consistent with those obtained by atomic absorption spectroscopy (AAS), confirming the accuracy and reliability of the proposed technique.*

Keywords: *Extraction, spectrophotometry, 1-butanol, Fe(III), azoreagent.*
DOI: 10.65382/2221-8688-2026-4-582-590

1. Introduction

Heavy metal ions released into soil and water pose major ecological and health risks [1-5]. They can be absorbed by humans and have been associated with oncological, cardiovascular, and neurological disorders [6]. Consequently, effective treatment of mine waters and industrial effluents is critical for minimizing environmental and public health hazards [7-10].

Iron is one of the most common contaminants. Even at low levels it affects the sensory properties of water, and higher concentrations contribute to pollution and physicochemical degradation of water bodies [11-13]. Reliable Fe(III) determination is therefore essential, and spectrophotometric methods remain widely used due to their sensitivity, selectivity, and operational simplicity [14-16]. Numerous chelating agents have been explored for Fe(III) analysis, offering high precision by transferring the analyte into an organic phase [17-23].

In this study, a newly synthesized bis-azopyrazolone reagent (R_1) was introduced, forming a stable, strongly colored complex with Fe(III) under mild conditions. Its extractive-spectrophotometric performance was validated using environmental water samples from the Zira district of Baku, demonstrating a selective, cost-effective, and practical method for monitoring iron contamination.

2. Experimental Part

2.1. Solutions and Reagents. All reagents were of analytical grade. $FeCl_3$ (10^{-3} M) and R_1 (2×10^{-3} M) solutions were prepared, with R_1 dissolved in ethanol. Buffer solutions (pH 1–6) were made using 0.1 M HCl, CH_3COOH, and NH_4OH. Extraction experiments employed

benzene, chloroform, carbon tetrachloride, and 1-butanol. Synthesis of the reagent is given in [24].

2.2. Extraction Procedure. A standard liquid–liquid extraction was performed by mixing 1.0 mL of Fe(III) solution with 2.0 mL of R_1 in a 25 mL flask and diluting to pH 5. The solution was extracted with 10 mL of organic solvent, shaken for 3 minutes, and allowed to separate for 1 minute. The organic layer containing the Fe(III)–R_1 complex was measured at 540 nm using a matching blank. All measurements were carried out in triplicate.

2.3. Application of the Proposed Method for the Determination of Fe(III) in Drinking Water Samples. A real water sample from the Zira district of Baku was analyzed to assess the method's practical applicability. After standing for 24 hours, 1 liter of the clarified water was acidified with nitric acid (1:1), evaporated to 70–80 mL, filtered, and diluted to 100 mL. Aliquots (1–3 mL) were treated with R_1, adjusted to pH 5, and extracted with 10 mL of 1-butanol, after which absorbance was measured at 540 nm. Fe(III) concentrations were obtained from the calibration graph. The results closely matched those from AAS, confirming the method's accuracy for trace Fe(III) determination in environmental samples.

2.4. Instrumentation. All UV-Vis measurements were carried out on Lambda-40 spectrophotometer (PerkinElmer, USA) using 1 cm quartz cuvettes. Solution pH was controlled with a PHS-25 ion-selective pH meter (Shaanxi, China). Quantitative analysis of Fe(III) in aqueous, extracted, and environmental samples was verified using an AAS-1N atomic absorption spectrometer (Carl Zeiss, Germany). Dielectric constants of the extraction media were determined with a Mettler-Toledo conductometer.

3. Results and Discussions

3.1. Effect of Extractants. The extraction performance of each solvent was assessed using two key parameters: the distribution coefficient (D) and the extraction factor (α). The distribution coefficient is defined:

$$D = \frac{C_{org}}{C_{water}}$$

The extraction factor (α), representing the fraction of the total analyte transferred to the organic phase, was calculated using the following expression:

$$\alpha = \frac{1}{\frac{1+V_{water}}{D \cdot V_{org}}}$$

Based on experimental data, the extraction factor was found to be α = 50, indicating highly efficient partitioning of the complex into the organic layer.

Among the solvents tested, 1-butanol provided the highest extraction efficiency (99.4%), remaining effective over wide phase ratios (10:1 to 200:10) and producing a complex stable for at least 2 hours and up to 50 °C, confirming good operational stability (Table 1).

Table 1. Effect of organic solvent on extraction efficiency of the Fe(III)-R_1 complex

Organic solvent	Dielectric constant	R,%	
1-butanol	17.8	99.4	
Chloroform	4.8	64.6	
Benzene	2.28	62.3	
Carbon tetrachloride	2.24	55.7	

3.2. Absorption Spectra of the Complex. Fe(III) formed a stable colored complex with R_1 at pH 5, enabling sensitive spectrophotometric detection [25]. The UV-Vis spectrum of the

extracted complex showed a distinct absorption maximum at 540 nm (Fig. 1).

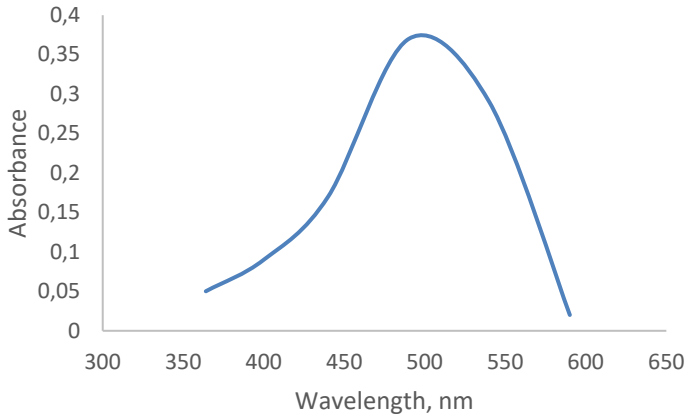

Fig. 1. Absorption spectra of Fe(III)-R_1 complex, $C_{Me}=10^{-3}$ M, $C_R=2\times10^{-3}$ M, l=1 cm

3.3. Effect of pH on the Extraction Efficiency of Fe(III)-R_1 Complex. The influence of pH on the formation and extraction of the Fe(III)–R_1 complex was studied to establish optimal conditions. Absorbance increased with rising pH and reached a maximum at pH 5, indicating the most efficient complex formation and extraction. At pH<4, protonation of the ligand's donor groups reduced its coordinating ability and lowered extraction efficiency [26, 27], whereas at pH>6, Fe(III) hydrolysis and the formation of $Fe(OH)_3$ decreased the amount of extractable complex. Therefore, pH 5 was identified as the optimum, providing maximal complexation while avoiding both protonation and hydrolysis, resulting in the characteristic bell-shaped profile shown in Fig. 2.

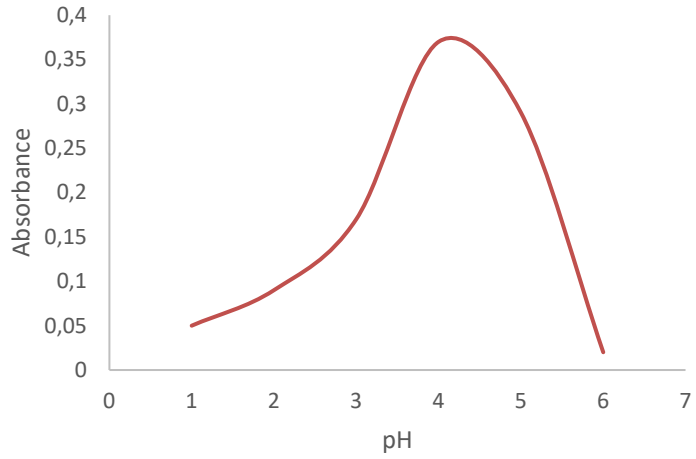

Fig. 2. Absorbance of Fe(III)-R_1 complex as a function of pH.
$C_{Me}=10^{-3}$ M, $C_R=2\times10^{-3}$ M, λ_{max}=540 nm, l=1 cm

3.4. Effect of Initial Fe(III) Ion Concentration on Extraction and Calibration Curve Construction. To evaluate the effect of initial Fe(III) concentration and construct a calibration curve, solutions containing 0.004×10^{-3} to 0.4×10^{-3} mol/L Fe(III) were prepared. Each was mixed with 2 mL of R_1 in 25 mL flasks and diluted to volume with pH=5 buffer to ensure optimal complexation and extraction. Absorbance was measured at λ_{max}=540 nm, and a calibration graph was constructed (Fig. 3).

The calibration plot exhibited excellent linearity over the range 0.112–3.36 μg/mL Fe^{3+}, confirming compliance with the Beer–Lambert law. A high correlation coefficient (R^2 = 0.983)

indicated strong analytical reliability. The calibration equation was y = 0.172x – 0.097, with all parameters summarized in Table 2. The calibration graph was evaluated with statistical treatments, including 95% confidence intervals for the slope (0.165–0.179) and intercept (-0.105–-0.089), ensuring precise calibration [28]. Standard deviations for each point ranged from 0.005 to 0.015, reflecting measurement variability. The standard error of estimate (S y/x) was 0.013, indicating minimal deviation from the fitted line, while the correlation coefficient (R^2 = 0.983) confirmed a strong linear relationship. These metrics validate the method's accuracy and reliability for Fe(III) determination.

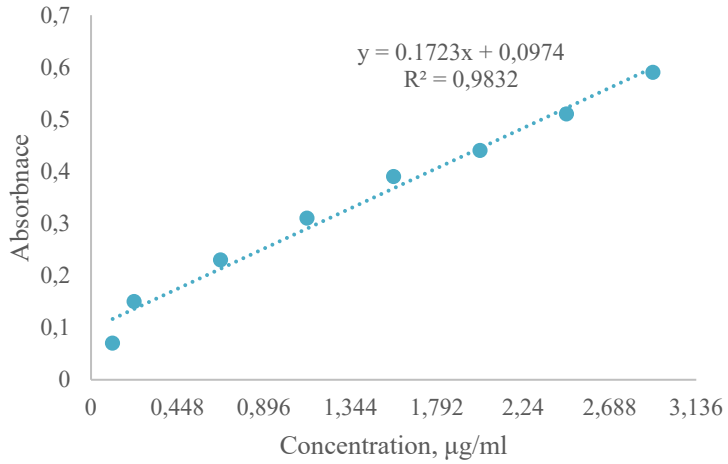

Fig. 3. Calibration graph showing adherence Fe(III)-R_1 complex to Beer–Lambert's law, $C_{Me}=10^{-3}$ M, $C_R=2\times10^{-3}$ M, λ_{max}=540 nm, l=1 cm

Table 2. Optimal Analytical Characteristics of the Extraction–Spectrophotometric Method for Fe(III) Determination

Parameter	Extractive spectrophotometric technique
Color of extracted complex	Reddish brown
Investigated pH range for extraction	1-6
Optimum pH for maximum extraction	5
Organic solvent used	1-butanol
Extraction time, (min)	3
λ_{max} (nm)	540
Molar absorptivity, ε (L/mol· cm)	6.1×10^4
Sandell`s sensitivity (mg/cm^2)	0.0049
Recovery (R, %)	99.4
Beer's law linear range (µg/mL)	0.112-3.36
Correlation coefficient (R^2)	0.983
Distribution coefficient (D)	124
Extraction factor	50
Detection limit (3σ) (µg/mL^1)	0.032

Table 3 provides detailed comparison of detection limits, molar absorptivities, extraction efficiencies, and selectivity for various methods.

Table 3. Comparative evaluation with other techniques.

Reference	Detection limit, µg/mL	Molar absorptivity, L·mol^{-1}·cm^{-1}	Extraction efficiency, %	Tolerance to interfering ions

Current method R_1	0.032	$6.1x10^4$	99.4	Excellent tolerance to Na(I), K(I), Ca(II), Ba(II), Cu(II), Cr(III) and others
[9]	0.2	$3.2x10^4$	95	Moderate tolerance
[3]	0.05	$5.0x10^4$	97	High selectivity, but moderate tolerance to multivalent ions
[25]	0.08	$6.5x10^4$	99	Good tolerance to alkali and alkaline earth metals
[19]	0.1	$5.5x10^4$	96	Moderate tolerance to Cu(II) and Cr(III)
[4]	0.15	$2.8x10^4$	92	Moderate tolerance to Na(I) and Ca(II)

3.5. Complex Formation and Stoichiometry Determination. The composition of the Fe(III)–R_1 complex was examined using the isomolar series method, Starik–Barbanel's relative yield method, and the equilibrium shift method. Astakhov's method [29] further confirmed that two protons are released during complex formation. A linear plot of $\lg\left(\frac{\Delta A}{\Delta A_{lim}-\Delta A)}\right)$ versus pH (4–5) yielded a slope of 2, consistent with the participation of $Fe(OH)^{2+}$ species and supporting the proposed complexation mechanism. The complexation of Fe(III) with the ligand (R_1) leads to a 1:2 Fe(III)-R_1 ratio, with Fe(III) predominantly existing as $Fe(OH)_2^+$ under the experimental conditions at pH 5. This coordination involves the azo group (-N=N-) and the pyrazolone ring acting as donor sites for the Fe(III) ion. The formation of the Fe(III)-R_1 complex is accompanied by a metal-to-ligand charge transfer transition, which is responsible for the characteristic absorption at 540 nm. The chromophore in this complex is likely the azo group, which is known to interact strongly with metal ions, imparting a stable, colored complex.

A structural diagram of the complex (Fig. 4) illustrates the coordination of the Fe(III) ion with two ligand molecules, each donating two donor atoms (e.g., nitrogen and oxygen).

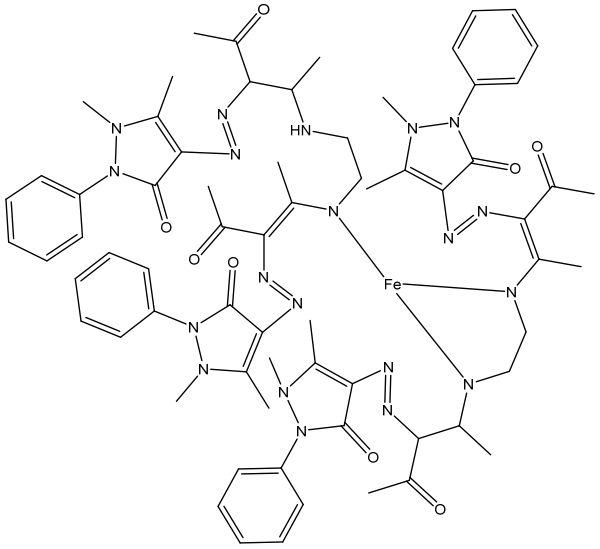

Fig. 4. The proposed structural diagram of the Fe(III)-R_1 complex

This 1:2 stoichiometry is consistent with other azo-pyrazolone complexes used for Fe(III) detection. Similar complexes, such as those reported by [9], also exhibit charge-transfer bands associated with the azo group. However, the R_1 ligand in this study differs by incorporating a pyrazolone ring, which may enhance the stability and selectivity for Fe(III) over other ions. Compared to other reagents, the Fe(III)-R_1 complex displays higher extraction efficiency and selectivity due to its unique structural properties.

3.6. Effect of Foreign Ions on Extraction of Fe(III) ions. The selectivity of the extraction method [30] was assessed by introducing various monovalent and multivalent cations at known excess levels and evaluating their effect on Fe(III) recovery using 1-butanol. The influence of these potentially interfering ions on extraction efficiency was then determined, with the results summarized in Table 4.

Table 4. Influence of Interfering ions on the extraction of Fe(III)

Ions	Tolerated Amount	Ions	Tolerated Amount	Ions	Tolerated Amount
Na(I)	2040	Cd(II)	5000	Sr(II)	750
K(I)	3010	Mn(II)	2590	Al(III)	650
Mg(II)	1500	Ni(II)	2700	Cr(III)	300
Ca(II)	250	Co(II)	2250	Ti(IV)	460
Ba(II)	8700	Cu(II)	480	Mo(VI)	810
Zn(II)	3000	Pb(II)	2000	W(VI)	450

The results (Table 4) show that the method exhibits excellent selectivity, with Fe(III) extraction remaining largely unaffected by a wide range of foreign ions. Alkali and alkaline earth metals, as well as transition and multivalent ions, caused no significant interference even at several thousand-fold excess. This high tolerance likely reflects the stability of $Fe(OH)^{+}$ species under the experimental conditions. The substantial differences in tolerance limits for various ions, such as the high tolerance for Ba(II) compared to Cu(II) and Cr(III), can be attributed to differences in their chemical properties. Ba(II) has a larger ionic radius and forms weaker complexes with the reagent, reducing its interference with Fe(III) extraction. In contrast, Cu(II) and Cr(III) have higher charge densities, leading to stronger complex formation and potential hydrolysis at pH 5, which interferes with Fe(III) extraction. The low tolerance for Cu(II) and Cr(III) may also be due to competitive complexation and the formation of insoluble hydroxides like $Cr(OH)_3$, which affect the extraction efficiency. Overall, the method is robust and well suited for Fe(III) determination in complex matrices containing multiple competing ions.

3.7. Determination of Fe(III) in Water Samples. The developed extractive spectrophotometric method was applied to environmental water samples and compared with standard AAS results (Table 5). The procedure involved forming the Fe(III)–R_1 complex, extracting it into 1-butanol under optimized conditions, and quantifying Fe(III) using a calibration curve. Five parallel measurements (n = 5) were evaluated at a 95% confidence level (P = 0.95), with mean values and confidence intervals calculated using Student's t-distribution.

Table 5. Results of Fe(III) determination in real water sample (comparison of proposed method with AAS, n=5, P=0.95)

Sample	Fe(III) content, mg/L	S_r	Comparative AAS result, mg/L	S_r
Water	1.29 ± 0.04	0.029	1.32± 0.01	0.045

4. Conclusion

In this study, a new pyrazolidinyl-based reagent (R_1) was synthesized and applied for the extractive spectrophotometric determination of trace Fe(III) in water. The reagent formed a stable Fe(III) complex that was efficiently extracted into 1-butanol, which provided the highest extraction efficiency (99.4%), with an extraction factor of 50 and a distribution coefficient of 124. Optimal complex formation occurred at pH 5, with maximum absorbance at

540 nm. The method obeyed Beer's law over 0.112–3.36 μg/mL and exhibited high molar absorptivity (6.1×10^4 $L\cdot mol^{-1}\cdot cm^{-1}$), Sandell sensitivity (0.0049 mg/cm²), and a low detection limit of 0.011 μg/mL. Stoichiometric analysis confirmed a 1:2 Fe(III)–R_1 complex, with Fe(III) mainly present as $Fe(OH)_2^+$. The method showed excellent selectivity toward common interfering ions and performed well in real water samples from the Zira district, giving results consistent with AAS. Overall, the study demonstrates that rational ligand design coupled with extraction techniques can yield highly sensitive and practical methods for environmental Fe(III) analysis.

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