Synthesis of New Dithiocarbamate Derivative Reagent for Micro Extraction and Determination of Iron(II) Ion in Aqueous Solution

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Abstract: *The new reagent N-methyl-N-((((4-chlorophenyl)diazenyl)thio) carbonothioyl)aniline (L) was synthesized via the reaction of sodium N-methyl-N-phenyldithiocarbamate with 4-chlorobenzenediazonium salt. The reagent was identified with many techniques, such as CHNS elemental analysis, UV-vis spectroscopy, FTIR spectroscopy, mass spectroscopy, ^{1}H-NMR, and ^{13}C-NMR spectroscopy. The Fe(II) ion was utilized to create a metal complex by reacting 2 mol of reagent with 1 mol of iron(II) chloride. Two approaches that were utilized for M:L ratio determination are mole ratio and Job methods. All of these refer to the 1:2 ratio for the complex. The solid complex was prepared and identified with the previous techniques, and additional electrical molar conductivity, and magnetic susceptibility measurements. A tetrahedral structure for the prepared complex was suggested based on spectroscopic measurements and elemental analysis results. Cloud point extraction was created for the preconcentration and separation of Fe(II) in drug samples by forming a rich phase using Triton X-114 at 80 °C temperature and pH 5 measurements utilizing flame atomic absorption spectroscopy and UV-vis spectrophotometer at λ_{max} 635 nm. The calibration curve of Fe(II) was linear in the extent of 0.2–1.0 µg/mL with r^2 of 0.996. The detection limit and quantification are 0.0504 and 0.1530 µg/mL.*

Keywords: *dithiocarbamate derivative; pre-concentration; cloud point extraction*

■ INTRODUCTION

Dithiocarbamate (DTC) is a soft donor reagent that can form chelates with the majority of the metal ions in the periodic table [1]. It is described as a "versatile ligand" and several researchers are concerned about DTC molecules because they have potential as helpful synthetic intermediates and protective groups in the synthesis of peptides and bonds in the organic solid phase [2]. They are organosulfur ligands that form stable complexes with metals [3]. There are numerous uses for DTC molecules and their derivatives, including antibacterial, antifungal, and anticancer properties [4-5]. Due to the possibility of functionalizing the substituents on the DTC backbone's nitrogen atom, which aids in the creation of numerous analogues of this chemical, DTC ligands are particularly helpful in complex synthesis. This has led to a variety of structures with improved characteristics, which therefore improve their physical and chemical properties [6]. These compounds are widely used as analytical reagents with significant environmental impacts because of their propensity to form complexes. Because of their great propensity to form chelates with metal ions at extremely low concentrations (g/mL), they are useful as extractive agents in the removal, preconcentration, and detection of hazardous heavy metal ions at trace and ultra-trace levels [7].

Cloud point extraction (CPE) is a modification of liquid–liquid extraction (LLE), a micelle-mediated separation method that is one of the most widely used techniques for pre-concentrating and separating organic molecules [8]. That is based on adding a surfactant solution at a certain concentration to the analyte sample and then adjusting the temperature or adding specific additives to move the analyte into a micelle phase to achieve separation [9]. The technique is simple, highly competent, concentrates well, inexpensive, safe, and

environmentally benign. Since CPE employs much less hazardous surfactants than harmful chemical solvents, it follows the principle of "green chemistry." Moreover, surfactants are not volatile or inflammable [9-10]. CPE readily combine with fluorescence [10], flow injection [11], flame atomic absorption spectroscopy (FAAS) [12] inductively coupled plasma mass spectrometry (ICP-MS) combines with FAAS, capillary electrophoresis of graphite furnace nuclear absorption spectrometry [13], UV-vis spectrophotometer [14-17], and high-performance liquid chromatography with fluorescence detection (HPLC-FLD) [18].

In this study, the preparation of a new dithiocarbamite derivative having azo-sulfur linkage was done. Then, the new reagent was used to synthesize metal complex with Fe(II), and successfully applied to extract and determine Fe(II) ion from various local pharmaceutical samples by reacting with the new reagent *N*-methyl-*N*-((((4chlorophenyl)diazenyl)thio)carbonothioyl)aniline by easy, low cost, and simple CPE coupling with UV-vis spectrophotometric method.

■ EXPERIMENTAL SECTION

Materials

The chemicals used in this project were obtained from Sigma, BDH, and Fluka. In this study, extremely pure materials were utilized. *N*-methyl-*N*-((((4-chlorophenyl)diazenyl)thio)carbonothioyl)aniline reagent (L) (0.01 mol/L) was prepared *via* dissolving 0.1609 g of reagent in 50 mL absolute ethanol. A stock solution of 1000 mg/L Fe(II) was prepared by dissolving 0.3560 g of $FeCl_2·4H_2O$ in 100 mL deionized water. A 0.1 mol/L NaOH was prepared by dissolving 0.4000 g in 100 mL. Also, 0.1 mol/L of 37% v/v HCl solution was prepared in 0.83 mL of 100 mL deionized water. Surfactants (20% v/v) of Triton X-100 and Triton X-114, and surfactants (1% w/v) of sodium dodecyl sulfate (SDS) and cetyltrimethylammonium bromide (CTAB) were prepared by taking 5.00 mL and 0.2500 g from each and diluting by deionized water in 25 mL. The following salts out were also prepared by dissolving 0.1 mol/L of NaCl, KCl, NaF, Na_2CO_3, Na_2SO_4, $(NH4)_2SO_4$, and NH_4Cl weighted 0.1461, 0.1863, 0.1049, 0.2649, 0.3551, 0.3303, and 0.1337 g, in 25 mL of deionized water,

respectively. Stock solution of interferences (1000 mg/L) was prepared by dissolving 0.1598, 0.4577, 0.3112, 0.3104, 0.2683, 0.3602, 0.1574, and 0.1630 g from the following $Pb(NO_3)_2$, $Cr(NO_3)_3$, $Ni(NO_3)_2$, $Co(NO_3)_2$, $CuCl_2·2H_2O$, $MnCl_2·4H_2O$, $AgNO_3$, and $CdCl_2$, respectively, in deionized water and diluted to 100 mL.

Instrumentation

A potassium bromide disc (4000–400 cm^{-1}) was utilized to record infrared spectra using an FTIR Shimadzu model 8400 equipment. ^{1}H-NMR spectra were recorded using a BAMX 400 MHz spectrometer in DMSO-d_6. Mass spectra were recorded using the mass spectrophotometer, Shimadzu LCMS 2010. A Shimadzu double beam 1800 UV was used to record UV-vis spectra, whereas CHNS was performed on an EuroEA Elemental Analyses. Estimating the melting points of compounds was done using the Gallen Kamp melting point. The molar conductivity for the iron(II) complex was measured using the Digital Conductivity Series. Magnetic susceptibility balance (Johnson Mattey) was used to calculate the magnetic susceptibility of iron(II) complex $[FeL_2]Cl_2$. Separating the aqueous phase from the rich phase was performed by the German Hettich centrifuge, model D-78532, and vortex mixer 945307 (USA). For all CPE studies, a water bath model BS-11 NO. K109.125 Korea was employed. WTW model 720 pH meter was used with a combined glass electrode and FAAS AA-6300 Shimadzu.

Procedure

Synthesis of sodium N-*methyl*-N-*phenyl dithiocarbamate*

First, 2 g of NaOH (0.05 mol) dissolved in a few deionized water in ice-cold temperature below 4 °C, after that 3.02 g of CS_2 (0.05 mol) and 5.44 mL of *N*-methylaniline (0.05 mol) was added. The mixture was stirred for 2 h. A yellowish-white solid product was formed, which was separated, filtered, washed with ether, and finally recrystallized from warm acetone [19]. The white solid product was dried under vacuum over $CaCl_2$ giving 1.68 g (yield = 84%), m.p. > 300 °C. CHNS: Found (calc.) %: C 46.80 (46.82), H 3.91 (3.92), N 6.80 (6.82), and S 31.22 (31.24).

Synthesis of N-*methyl*-N-*((((4-chlorophenyl)diazenyl) thio)carbonothioyl) aniline (L)*

The *p*-chloroaniline weighted 0.9185 g (0.0072 mol) was dissolved in 10:10 mL of HCl/H$_2$O and the mixture was vigorously stirred while it cooled to a temperature of 0 to 5 °C. To this solution, 0.5000 g of NaNO$_2$ (0.0072 mol) dissolved in 3.00 mL of deionized water was added drop by drop and continued stirring in the ice bath at 0 to 5 °C. After that, a black solution was noted after 30 min of stirring. The black solution was added drop by drop to the smallest amount of the aqueous solution containing 1.5000 g of sodium *N*-methyl-*N*-phenyldithiocarbamate (0.0072 mol), and the mixture was stirred for 1 h at 0 to 5 °C. The resulting brown precipitate was filtered and washed under cold water. Yield 76%, m.p. 123 °C. CHNS: Found (calc.) %: C 52.25 (52.17), H 3.76 (3.66), N 13.06 (13.002), and S 19.92 (19.80). Scheme 1 illustrates the procedures for reagent preparation.

Synthesis iron(II) complex

To the 5 mL ethanolic solution of the FeCl$_2$·4H$_2$O (1 mol), an ethanolic solution of the reagent L (2 mol) was added, and the mixture was refluxed for 1 h. The yellowish-green formed precipitate was filtered, and washed with ethanol, and dried under vacuum. Yield 64%, m.p. 133 °C.

Sample preparation

The procedure for selecting thirteen tablets from three different manufacturers was applied for commercially available tablets. Each brand's tablets were weighed, then mortar-crushed and ground into a fine powder. A 1000 μg/mL solution for Fe(II) was prepared by weighing an amount equal to the active component of each pharmaceutical preparation. In deionized water, the powder was dissolved. After that, it was filtered to get rid of any undissolved residue that might have impacted the response. The filtrate was completed to 100 mL. Further dilution was necessary to allocate the concentration within the linearity of the calibration graph Table 1 shows samples of Fe(II).

CPE procedure

According to the CPE procedure, aliquot of 10 mL of 10 μg/mL of iron(II) was added to 0.5 mL of 0.01 mol/L of reagent, 0.5 mL of 20% v/v Triton X-114 was then added, in a vortex mixer thoroughly mixed the solution. Place the tube in the 80 °C water bath for 10 min. Centrifugation at 4,000 rpm for 5 min was used to separate the phases. The aqueous phase was simply removed by decantation. The colored product's surfactant-rich phase was dissolved in 0.5 mL of ethanol, and the product's absorbance was measured at λ$_{max}$ 635 nm in comparison to a blank reagent made under
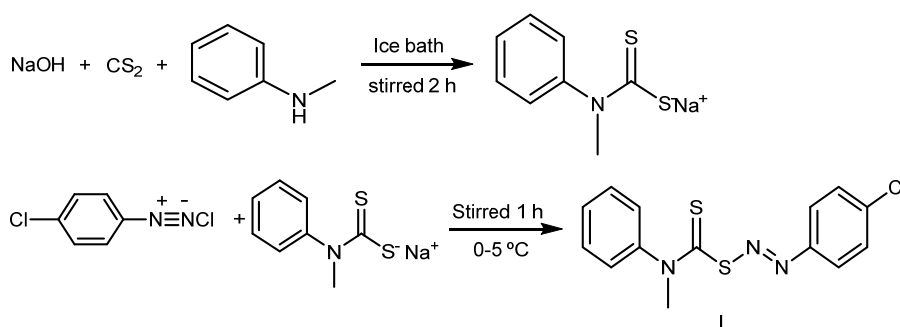
Scheme 1. Illustration of the preparation of L

Table 1. Pharmaceutical samples of Fe(II)

Sample number	Pharmaceutical drug company and claimed content of active ingredient	Average wt (g) of tablet (n = 13)	wt of sample (g) of active ingredient to obtain 1000 ppm in 100 mL
B1	Ferrous Sulfate, Accord, Barnstaple, 65 mg	0.4423	0.6804
B2	FeroFix, Pioneer, Iraq, 47 mg	0.3460	0.7361
B3	Iron, Mary Care, Holland, 49 mg	1.2867	2.6259

optimum conditions. The proposed mechanism of Fe(II)-L complex separation by CPE is illustrated in Scheme 2. Some equations used in the extraction are shown in Eq. (1-5).

Distribution ratio (D)

$$= \frac{\text{concentration of analyte in rich phase}}{\text{concentration of analyte in aqueous phase}} \quad (1)$$

$$\text{Extraction percent (E\%)} = \frac{D}{1+D} \times 100 \quad (2)$$

$$\text{Recovery (\%)} = \frac{\text{Measured value}}{\text{Actual value}} \times 100 \quad (3)$$

$$\text{Relative error percentage (Ere\%)} = \frac{\text{Absolute error}}{\text{Actual value}} \times 100 \quad (4)$$

$$\text{Absolute error} = \text{Actual value} - \text{Measure value} \quad (5)$$

■ RESULTS AND DISCUSSION

Characterization of New Synthetic Reagent

FTIR spectroscopy

The prepared L reagent contains importance groups such as NCS_2 and $-N=N-$. The absorption band belonging to C–N of NCS_2 appeared at 1485 cm^{-1} [20]. The $\nu(CS_{2\,\text{asym.}})$ and $\nu(CS_{2\,\text{sym.}})$ observed at 1006 and 1091 cm^{-1}, respectively [21]. Furthermore, the peaks at 1589 and 787 cm^{-1} were ascribed to the $\nu(-N=N-)$ and $\nu_{\text{asym}}(Cl)$ stretching vibrations respectively [5,22]. When complexing with the Fe(II) ion, the absorption bands of these groups changed. Thioureide $\nu(C–N)$ band was detected at 1465 cm^{-1} [23], whereas the $\nu(C–S)$ stretching
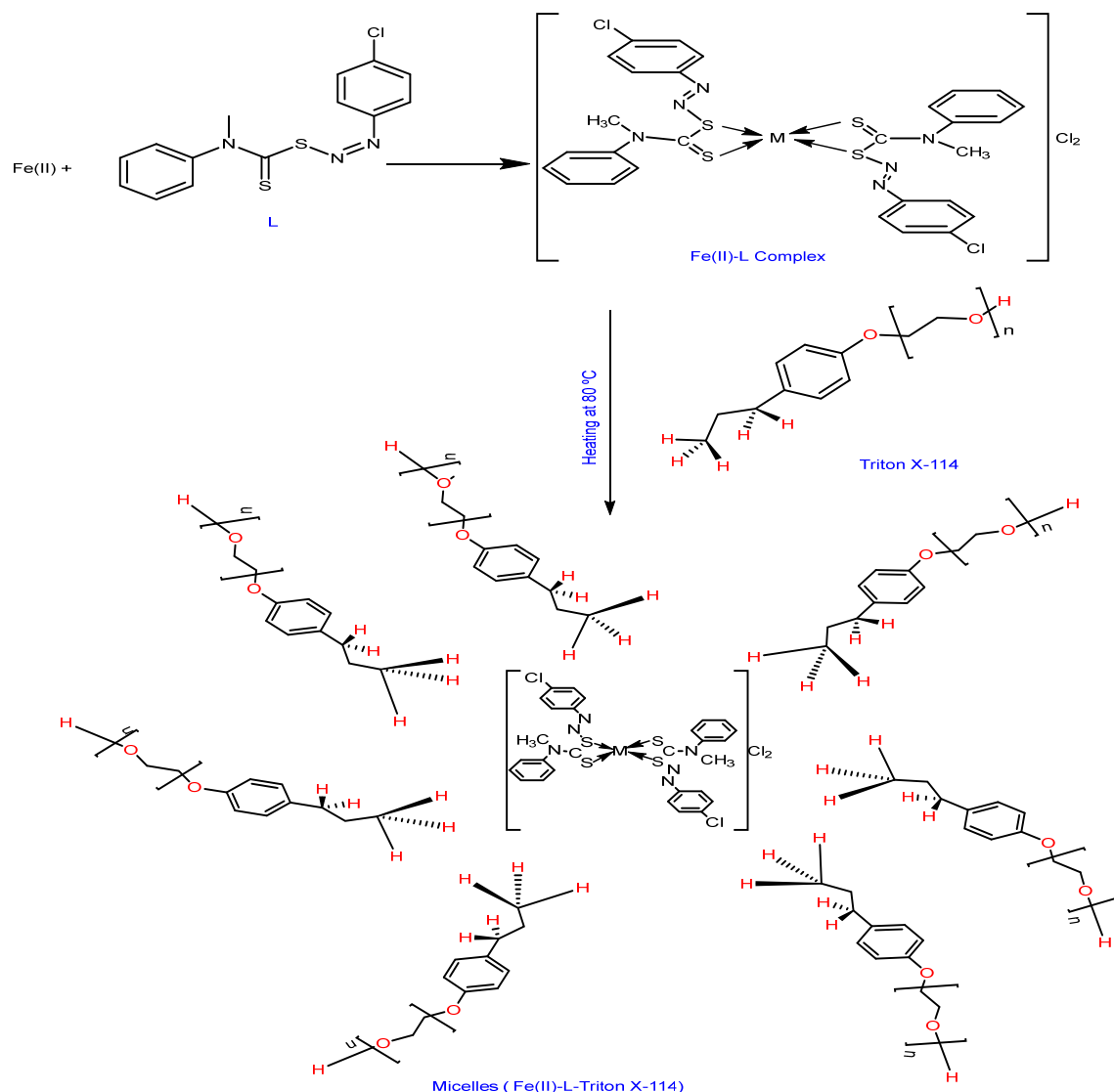
Scheme 2. Proposal mechanism of Fe(II)-L complex separation by CPE method

showed at 1168 cm^{-1}. The azo functional group stretched at 1590 cm^{-1}. The C=S characteristic stretching frequency vibrated at 1091 cm^{-1} [20]. Moreover, additional band of have been noted at 497 cm^{-1} related to ν(Fe–S).

Mass, ¹H-NMR, and ¹³C-NMR spectroscopy

The mass spectra are used to measure the mass-to-charge ratio (*m/z*) of the reagent L and its complex. The experimental parent molecular ion peaks of the reagent and its complex at 321.5 and 770.2 *m/z*, respectively. As the most significant reagent are azo, thioureido, and thiocarbonyl coupled with the production of positive molecular ions and other positively charged fractions,

because of their significant relative abundance, several fractionation paths were used for the reagent and complex [24]. The mass spectra of prepared compounds are noted in Fig. 1.

The ¹H-NMR spectrum of L in Fig. 2 showed a singlet peak at δ = 3.37 ppm equal to the methyl group's three protons in N–CH₃. A number of signals were seen in the δ region of 7.02–7.33 ppm are assigned to *N*-phenyl rings protons as follows: at 7.02–7.06 ppm as a triplet peak corresponds to the aromatic proton, at 7.25–7.27 ppm as a triplet peak corresponding to the aromatic proton in both places and at 7.31–7.33 ppm as a doublet
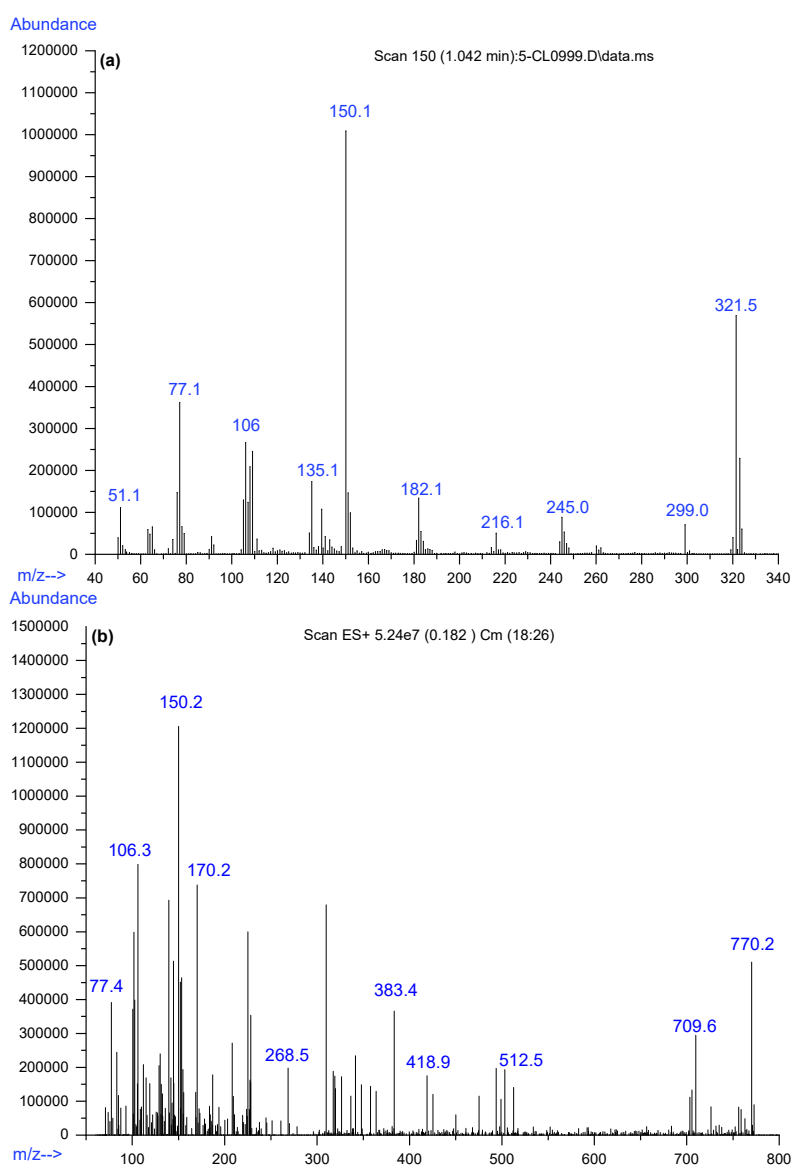

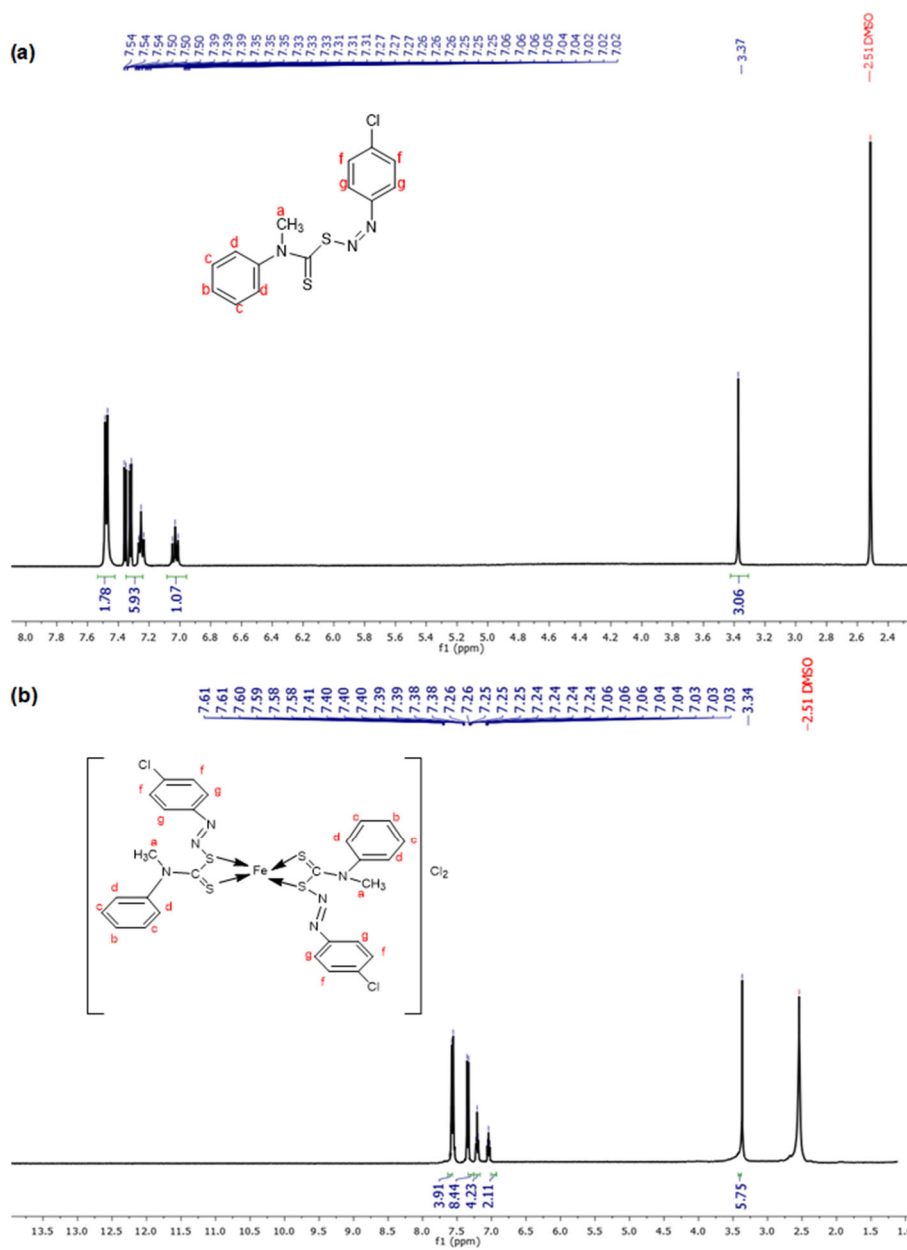**Fig 1.** Mass spectrum of the (a) L and (b) Fe(II) complex

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Fig 2. ^{1}H-NMR spectrum of the (a) L and (b) Fe(II) complex

peak corresponding to the aromatic proton in both places [5]. This indicates that the substitution took place on the sulfur atom during the reaction between the diazonium salt and sodium *N*-methyl-*N*-phenyldithiocarbamate to produce L. Several of the signals seen in the area of 7.35–7.54 ppm is assigned to azo-phenyl rings protons [25]. The coordination of L to the Fe(II) ion caused the most of proton signals (Fig. 2(a) and (b)) to change and shift during complexation. A singlet peak at 3.43 ppm belongs of the methyl group of N–CH$_3$. The numerous signals

seen in the area of 7.03–7.26 ppm are assigned to the *N*-phenyl rings' protons [5]. Peaks at 7.38–7.61 ppm is attributed to azo-chloro benzene protons [26-27].

The ^{13}C-NMR spectra of L and its complex with Fe(II) in Fig. 3(a), the thiocarbonyl carbon (C=S) signal at site 193.94 ppm in a weaker field, which is a representation of the DTC substituent in the ^{13}C-NMR spectrum [19]. Signals in the range of 39.37–40.62 ppm attributed to methyl carbon attached to the nitrogen atom [28]. Singlet peaks of the ligand were shown at

120.05, 125.75, 127.44, 129.36, 135.52, 140.01, and 157.60 ppm which belong to carbons of aromatic rings. The ^{13}C-NMR spectrum of the complex in Fig. 3(b), was recorded in DMSO-d_6, in comparison to the free ligand, 40 ppm up field shifts are shown for all of the complexes for NCS_2 carbon. This further supports the coordination by CS_2.

Absorption spectra

The electronic spectra for L and its complex are shown in Fig. 4, the spectrum of the ligand exhibited band at 411 nm for n→π* transition NCS_2 and –N=N– group.

While the complex has λ_{max} 635 nm corresponding to charge transition [29-30].

Enhancing the Experimental Conditions

In order to improve the method's sensitivity and to promote the extraction effectiveness for CPE, the most significant parameters effects are optimized using the traditional optimization technique. The parameters including pH effect, order of addition, volume of surfactant effect, reagent concentration, surfactant types, temperature effect, time of heat, salt out, and interfering effect.
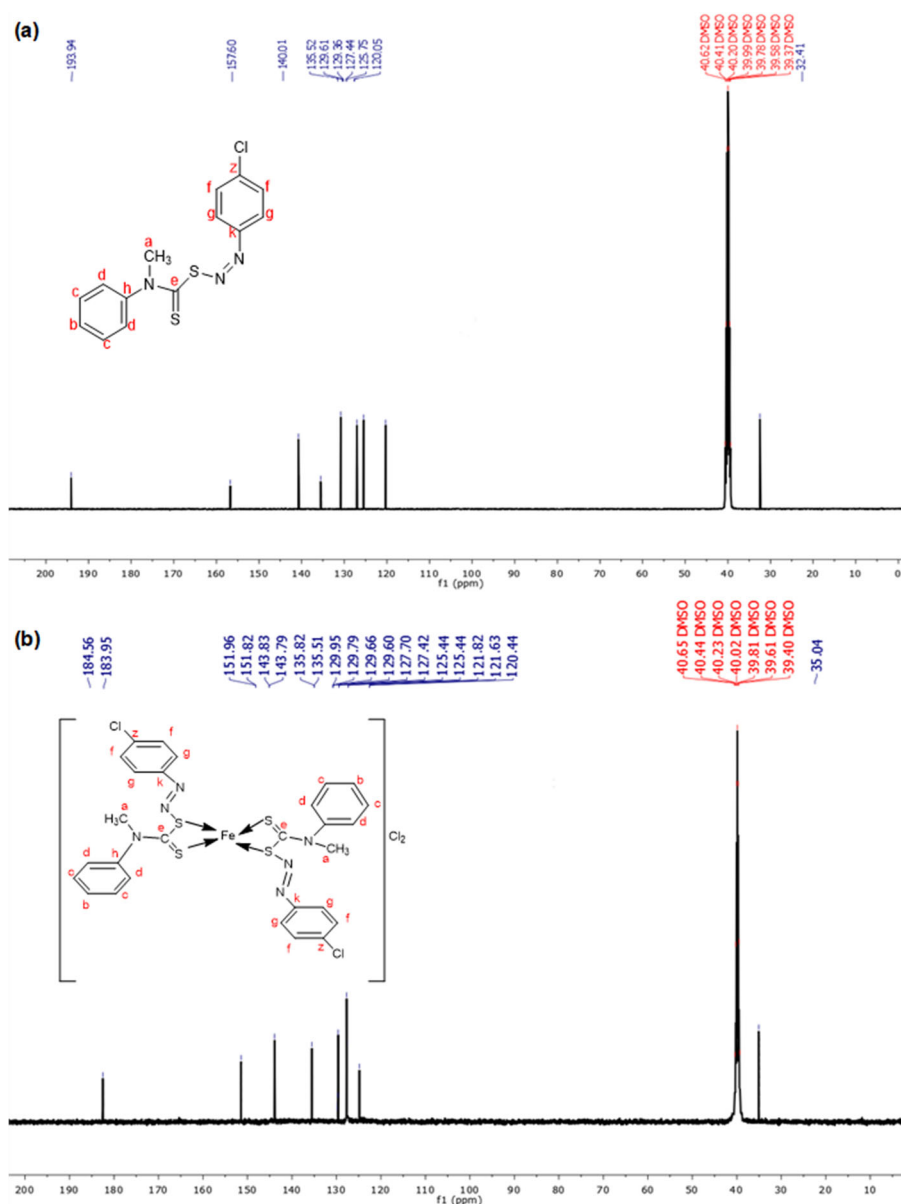

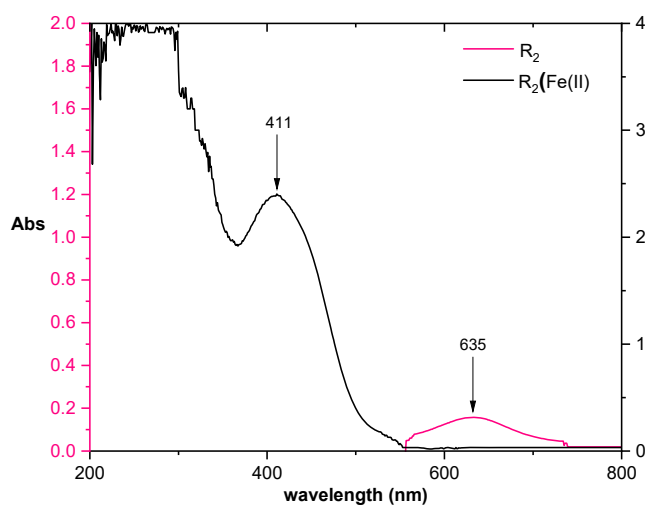**Fig 3.** ^{13}C-NMR spectrum of the (a) L and (b) Fe(II) complex

Fig 4. Absorption spectra for L and Fe(II) complex

Order of addition

The most important factors studied are the addition order. A change of order was of iron complex formation by using (10 μg/mL, 10 mL) of Fe(II) and reagent (0.01 mol/L, 0.5 mL) with 0.5 mL of 20% v/v Triton X-114. then heated for 5 min temperature of 75–80 °C then separated by using a centrifuge, then add 0.5 mL of ethanol to the rich phase for solubility after that measured by UV-vis the better absorption outcome as the sequence addition L-Fe(II)-Triton X-114 were chosen.

Reagent concentration and pH effect

This parameter plays a critical role in the formation of metal complex and subsequent extraction, and it has been an important factor for CPE. In this work, the pH impact was studied within a pH range of 1–10 by using pH solutions of 0.1 mol/L HCl and 0.1 mol/L NaOH, and the outcomes were shown in Fig. 5. At pH above 5, the absorbance reduces due to the formation of hydroxide of iron ion.

The concentration of the ligand is one of the main elements influencing the effectiveness of extraction. Various concentrations of the reagent in the extent 0.8×10^{-3} to 4.5×10^{-3} mol/L were utilized. The outcomes are shown in Fig. 6. The better results are 3.5×10^{-3} mol/L of reagent.

Surfactant kind and Triton X-114 concentration effect

The type of surfactant utilizes depends on the CPE efficacy. In this study, using specific concentrations of various types of surfactants, like Trition X-100, Trition X-114, CTAB, and SDS. When utilizing separate surfactant kinds, it was found that utilizing Triton X-114 produced the best results, as shown in Fig. 7(a). One of the non-ionic surfactants used frequently in CPE is Triton X-114. Because it has benefits such as high commercial purity, low toxicity, low cost, high density of the surfactant-rich phase enabling the phase separation by centrifugation,
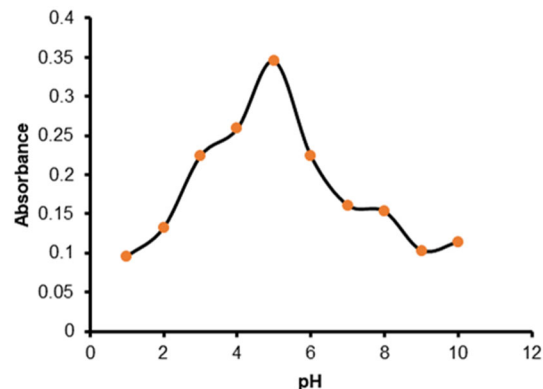

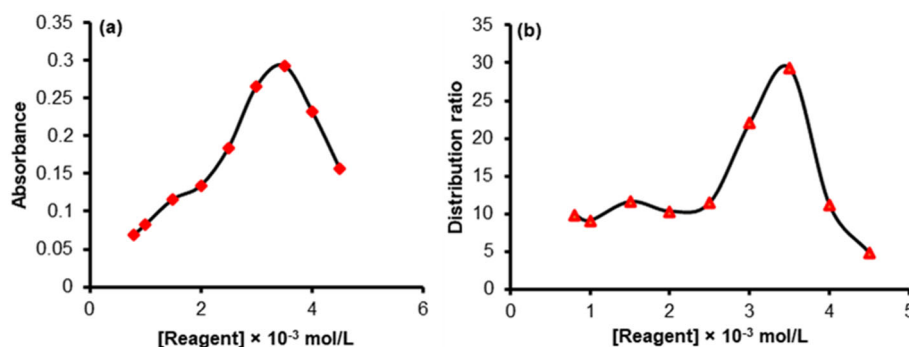**Fig 5.** Effect of the pH for Fe(II)complex

Fig 6. Effect of concentration reagent (L) on Fe(II) complex: (a) variation of reagent concentration effect and (b) distribution ratio estimation
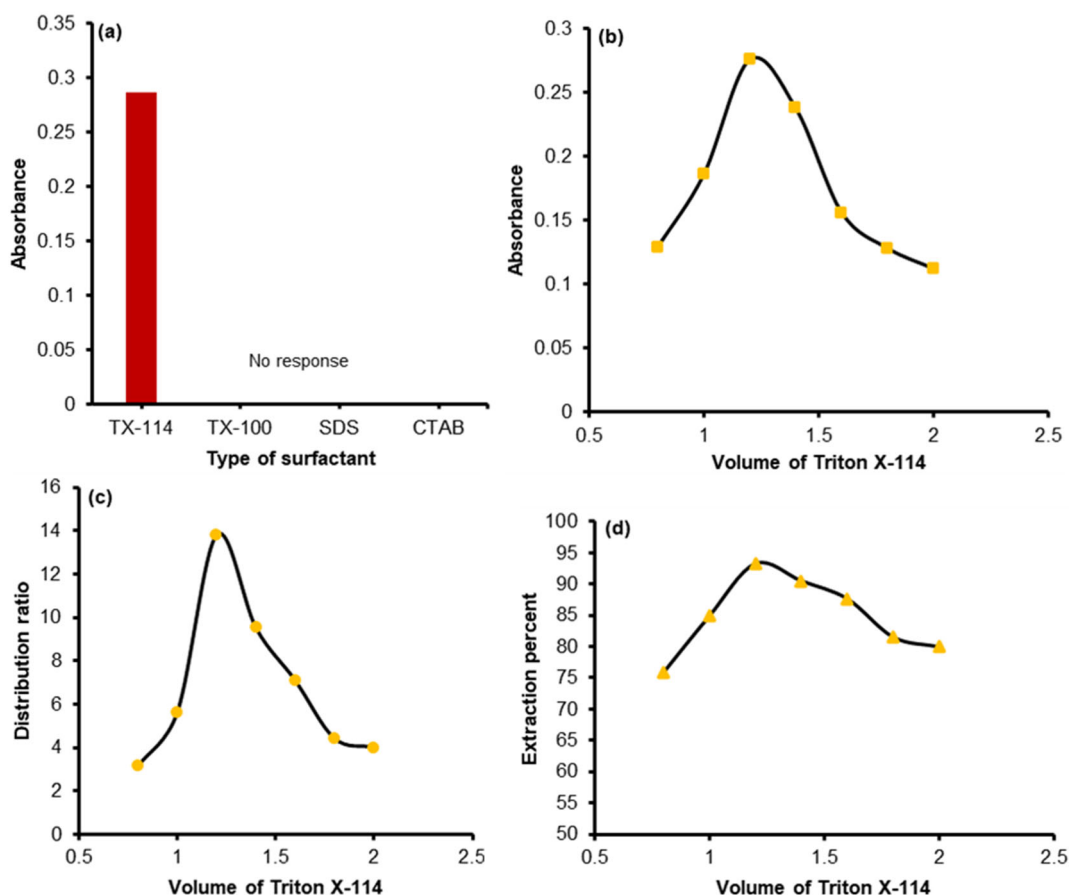
Fig 7. Surfactant effect of Fe(II) complex: (a) type of surfactant, (b) variation volume of surfactant, (c) distribution ratio, and (d) extraction percentage

and comparatively low cloud point temperature. The impact of non-ionic surfactant amount of 20% v/v of Triton X-114 of 0.8–2.0 mL. When the amount of Triton X-114 was increased, it was found that the complexes' absorbance increased until 1.2 mL, as shown in Fig. 7(b-d).

Incubation time and temperature effect

The effect of the equilibrium temperature is studied because they are crucial for the successful completion of the reaction and the efficient separation of the phases. The temperature between 50 and 85 °C was examined. The optimal temperature for all later measurements was chosen at 80 °C as shown in Fig. 8(a). An investigation of the incubation time between 5 and 30 min was conducted, and the analytical signal was entirely dependent on the incubation time. Incubation time of 10 min was chosen as an appropriate amount of time for the later

measurements, as shown in Fig. 8(b).

Salt out and interference effect

The extraction process may be impacted by the solution's addition of salt. The impact salt was studied on CPE by adding 0.5 mL of 0.1 mol/L of KCl, NaCl, NaF, NH_4Cl, $(NH4)_2SO_4$, Na_2CO_3, and Na_2SO_4 to the solution after completion of the method product acquired show. All salts were seen to slightly decrease, as shown in Table 2.

Interference's impact expected was studied to know the approach selectivity under study by adding 0.5 mL from each interference of $Ni(NO_3)_2$, $Pb(NO_3)_2$, $CuCl_2·2H_2O$, $Cr(NO_3)_3$, $Co(NO_3)$, $MnCl_2·4H_2O$, $CdCl_2$, and $AgNO_3$ (0.1 mol/L) with 10 mL of 10 ppm Fe(II). Noted that all interference interferes with metal and causes an increase in absorption, as shown in Table 3.
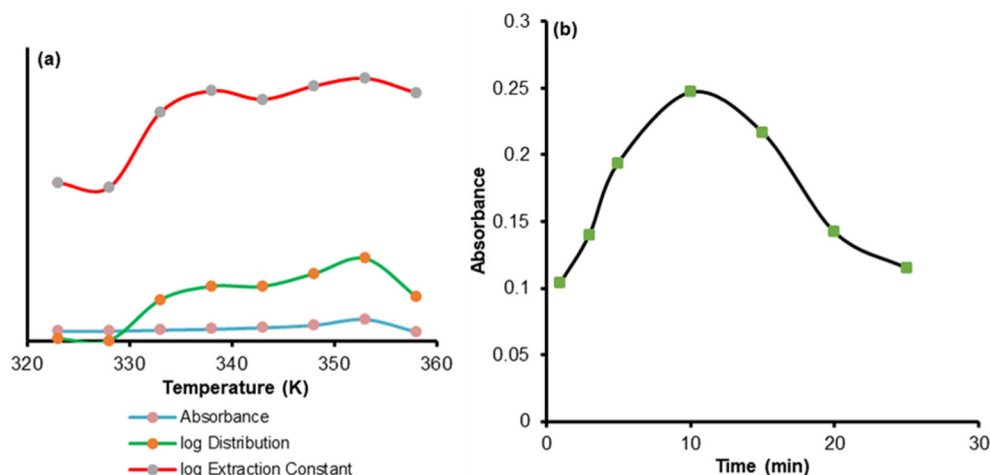
Fig 8. (a) Overlapping between Abs, log D, log K_{ex} vs temperature of Fe(II) complex and (b) time effect of Fe(II) complex

Table 2. Effect of salt out for Fe(II) complex

Salt out	Abs filtrate	Abs rich phase	D	E%	Recovery%	Ere%
Abs. without	0.010	0.249	24.900	96.139	17.351	82.648
NaCl	0.013	0.115	8.846	89.843	46.184	53.815
KCl	0.011	0.202	18.363	94.835	175.652	75.652
NaF	0.007	0.093	13.285	93.000	46.039	53.960
Na_2CO_3	0.008	0.135	16.875	94.405	145.161	45.161
Na_2SO_4	0.012	0.101	8.416	89.380	74.814	25.185
$(NH4)_2SO_4$	0.011	0.150	13.636	93.167	148.514	48.514
NH_4Cl	0.013	0.117	9.000	90.000	78.000	22.000

Table 3. Effect of interference Fe(II)complex

Interfering ion	Abs filtrate	Abs rich phase	Recovery%	Ere%
Abs. without	0.010	0.249	18.568	81.431
$Pb(NO_3)_2$	0.011	0.122	48.995	51.004
$Cr(NO_3)_3$	0.024	0.116	95.081	4.918
$Ni(NO_3)_2$	0.009	0.083	71.551	28.448
$CdCl_2$	0.010	0.112	134.939	34.939
$Co(NO_3)_2$	0.010	0.126	112.500	12.500
$CuCl_2 \cdot 2H_2O$	0.044	0.291	230.952	130.952
$MnCl_2 \cdot 4H_2O$	0.012	0.090	30.927	69.072
Ag NO_3	0.025	0.215	238.888	138.889

Stoichiometry of the complex

Two approaches were used to determine the complexes' stoichiometry. First, by using the Jobs method, solutions of 1×10^{-3} mol/L reagent and 1×10^{-3} mol/L of Fe(II) solution were prepared and mixed in continuous variation then measuring the solution by UV-vis spectrophotometer at 635 nm to be 1:2, Fig. 9(a) shows Job's plot of the complex. Second by using mole ratio method, whereas in this method a solution of 1×10^{-3} mol/L reagent and 1×10^{-3} mol/L of Fe(II) solution were prepared, the size of Fe(II) solution was steady and the reagent size changed, or the opposite, then measured using a UV-vis spectrophotometer at 635 nm after being diluted to 10 mL of ethanol. The stoichiometry of the compound was determined to be 1:2. Fig. 9(b) shows the mole ratio plot of the complex.
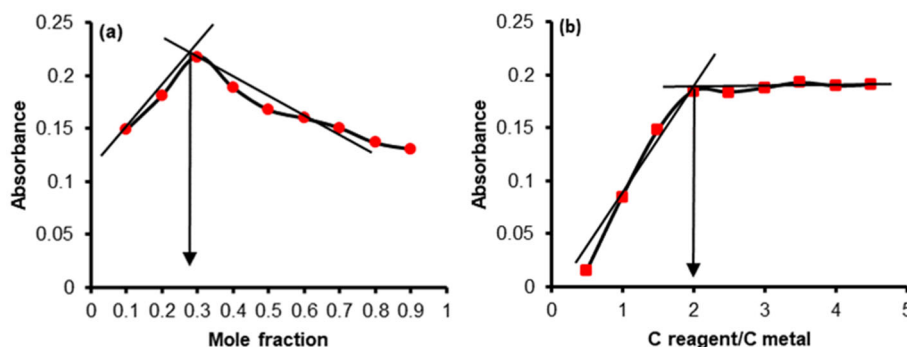
Fig 9. Stoichiometry of Fe(II) complex (a) Jobs method and (b) mole ratio method

Calibration curve

Under the predetermined optimal circumstances, by adjusting the metal ion concentration at range from 0.2–10 μg/mL, the Fe(II) calibration curve was assessed. It was shown that absorption rises with increasing concentration as it is a linear line. Absorbance and concentration were plotted in the calibration curve. Table 4 shows the results of the linear regression equation, linearity, slope, intercept, relative standard deviation (RSD%), correlation coefficient (r), coefficient of determination (r^2), limit of quantification (LOQ), and limit of detection (LOD) for

the estimation of Fe(II) using CPE and comparison with other published methods. It was found that the new method has high sensitivity and can be used to estimate Fe(II) in the other samples.

Application

After the metal study was completed of metal was studied on many medication models of drug taken from were taken and was the sampling process and the drug's content was assessed spectrophotometric at λ_{max} 635 nm. By utilizing the t-test and F-test to compare the results of

Table 4. Comparison of the CPE-UV-vis method for estimating Fe(II) with previously published methods

Parameters	CPE-UV-vis new method	CPE-UV-vis published method [31]
Regression equation	y = 0.117x + 0.028	y = 0.0089x + 0.018
r	0.998	0.998
r^2	0.996	0.996
Linear range (ppm)	0.2–1.0	5–112
LOD (ppm)	0.050	0.800
LOQ (ppm)	0.153	-
Slope	0.117	0.008
Intercept	0.028	0.018
RSD% (n = 5)	1.376	2.000

Table 5. Statically data pair t-test for the determination of Fe(II) in pharmaceutical drugs

No. of sample	Quoted value and active ingredient	Practical content (conc.) mg/L		X_d (mg/L) New method with FAAS	$\overline{X}_d$ (mg/L) ± S_d New method	Paired t-test $\overline{x}_d\sqrt{n}/\overline{s}_d$ New method	F_{cal}
		New method	FAAS method				
B1	65 mg, (1 mg/L)	0.95	1.02	−0.07			
B2	47 mg, (1 mg/L)	1.10	1.01	0.09	0.086 ± 0.155	0.967	15.00
B3	49 mg, (1 mg/L)	1.20	0.96	0.24			
	($\overline{X}$ ± σ_{n-1})	1.08 ± 0.125	0.99 ± 0.032				

Value of $F_{tab.}$ at 95% confidence level, d.f $_{(3,3)}$ =19.00 and t_{tab} at 95% confidence level, d.f$_{n-1}$ = 4.303, $F_{cal.} < F_{tab.}$ non sig. difference, $t_{cal.} < t_{tab}$ non sig. difference

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the new approach with a stock FAAS method for determining Fe(II) in the sample solutions. It was found that the concentration did not match the quoted value. Therefore used the t-test and F-test, where appears that there is no significant difference between the new method and the FAAS method, as shown in Table 5.

■ CONCLUSION

Reagent L was utilized to prepare FeL complex. According to experimental data, complexes' tetrahedral structural formula was proposed. The molar conductance values of the FeL complexes indicate a 1:2 electrolytic natures. A CPE which was ecofriendly, sensitive, and selective spectrophotometric approach was done for determining low concentration of Fe(II) using L reagent as complexing agent. The effectiveness of CPE is affected by pH at 5, 20% triton X-114, incubation time 10 min, and 80 °C of heating. The suggested method was effective, the potential advantages of the current method are time savings, reducing the quantity of reagents used as well as minimizing analyst effort. This method has been successfully applied to drugs such as Iron, FeroFix, and Ferrous Sulfate, and compared with FAAS method, there were no significant differences, and this indicates the possibility of using it as an alternative method.

■ ACKNOWLEDGMENTS

■ CONFLICT OF INTEREST

The authors do not have conflict of interest.

■ AUTHOR CONTRIBUTIONS

Ivan Malik Shaker, Ahmed Fadhil Khudhair, and Hayder Hamied Mihsen contributed to the design and implementation of the research, to the analysis of the results and to the writing of the manuscript. All authors agreed to the final version of this manuscript.

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