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Metal Complexes Of The Schiff Base Of 1H-Indole-3-Carbaldehyde Were Produced And Their Catecholase Activities Were Assessed

Hiyam Hadi Alkam[1] , **Eman Mutar Atiyah**[2], **Rehab Kadhim Al-Shemary**[3]

[1,3]Department of Chemistry, College of Education for Pure Science, Ibn Al- Haitham, University of Baghdad, Iraq

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Abstract: ThisTheSchiff base 4-(((E)-(1H-indol-3-yl)methylene)amino)-6-(((E)-indolin-3-ylmethylene)amino)benzene-1,3-dithiol was synthesized and characterized in this paper, which included ^{1}H NMR spectroscopy. In the Schiff base reaction that led to the formation of the ligand, both ends of azmothine were involved. Four metal complexes [Cu(II) and Fe(III)] with chloride and thiocyanate groups (SCN–) in N- and N-indole ,N-azmothine and S-bonding modes were studied for their ability to mimic catecholase oxidase. All of the metal complexes are catalytically active, with complex3 having the highest turnover rate (kcat). The formation of H_2O_2 was revealed by the catalytic process as monitored by ^{1}H NMR spectroscopy (isolation of 3, 5-DTBQ) and iodometric titration, implying that the mechanism of oxidation is through the formation of a semiquinolate species.

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1. Introduction

Lately, in order to slow the spread of drug resistance, new antimicrobial agents with chemical properties that differ markedly from those of existing ones have been designed and synthesized. Because of their synthetic and biological importance, indoles and their fused heterocyclic derivatives have received a lot of attention, such antitumor as analgesic [1, 2], anticancer [3, 4], [5, 6], antimicrobial [5–7], anticonvulsant [8, 9], and antiproliferative activities [10]. Schiff bases, which are derived from indoles, exhibit analgesic, anti-inflammatory,antidepressant, antimicrobial, and properties [13].When bioorganic molecules or drugs are bound to metal ions, their biomimetic properties, therapeutic effects, and pharmacological properties change dramatically. Metal complexes of Schiff bases derived from indoles have been synthesized, and it has been observed that antimicrobial activity of the Schiff bases is significantly indoles ligands have spin-crossover properties and can be used as molecular-based memory devices, display and optical switches [16, 17]. Keeping in view medicinal and industrial applications of indoles and potential chemistry of transition metals;Metal complexes of Schiff bases derived from indoles have been synthesized, and it has been observed that antimicrobial activity of the Schiff bases is significantly indoles ligands have spin-crossover properties and can be used as molecular-based memory devices, display and optical switches [16, 17]. Keeping in view medicinal and industrial applications of indoles and potential chemistry of transition metals

Experimental

Materials and physical measurements

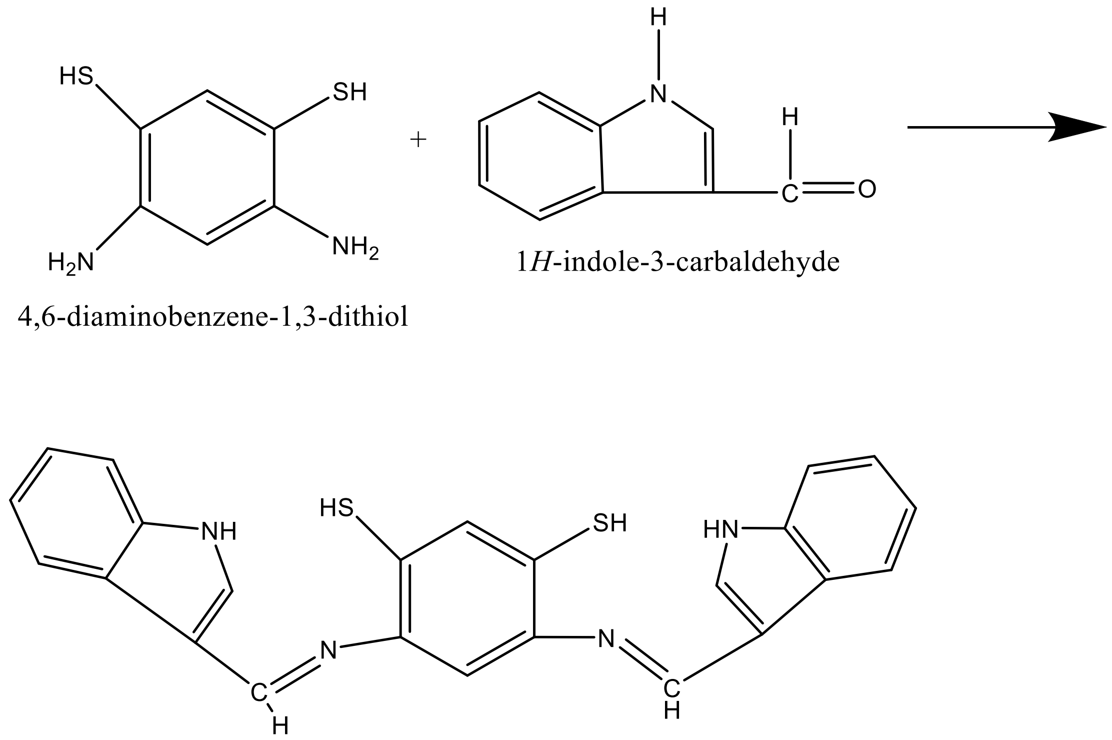
4,6-diaminobenzene-1,3-dithiol, 1H-indole-3-carbaldehyde, DMSO-d6, potassium thiocyanate, and 3, 5-di-tertbutylcatechol were all purchased and used from Sigma Aldrich. Sigma Aldrich provided the chloroform, isopropanol, andmethanol. All chemicals and solvents were analytical grade and were used without purification as received. $CuCl_2.2H_2O$ (copper(II) chloride dihydrate) and $FeCl_3.6H_2O$ (iron(III) chloride hexahydrate) were the hydrated metal salts used. An ElementarAnalysensystemeVarioMICRO was used to perform elemental analysis (C, H, andN).

Synthesis of the Ligand

To make the Schiff base, an ethanol solution of 4,6-diaminobenzene-1,3-dithiol (1mmol in 20cm-3) was refluxed for 5 hours with an ethanol solution of 1H-indole-3-carbaldehyde (1mmol in 15cm-3) After the reaction mixture was cooled, the formed product was filtered off, recrystallized from ethanol, washed with diethyl ether, and dried in a vacuum desiccator over silica gel. (see Scheme 1).

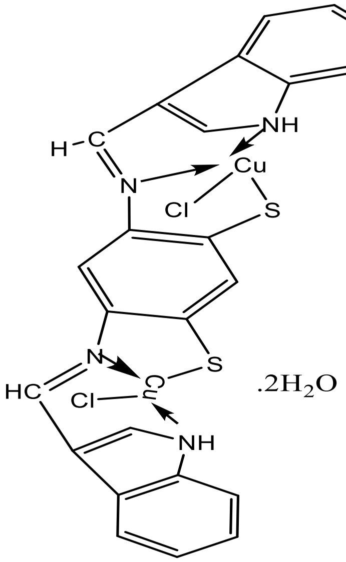
Metal complexes are synthesized

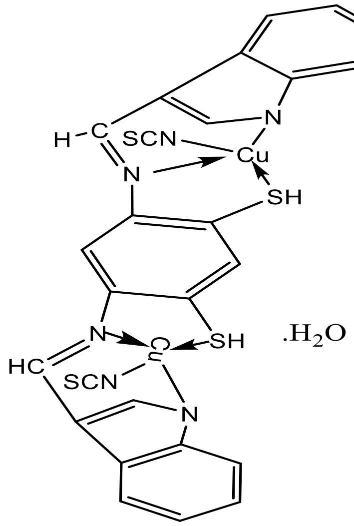
A general procedure was used to make the four metal complexes. An equimolar 5 mmolmethanolic solution of the hydrated metal-chloride salt was added to a 5 mL warm chloroform solution of the ligand (5 mmol).In order to make the thiocyanate metal complex, an equal mole of potassium thiocyanate dissolved in methanol was added. A few drops of triethylamine were also added to the Fe(III) complexes as a deprotonating agent during the synthesis. The resulting mixture was stirred for 6 hours at room temperature, then filtered and washed twice with a 1:1 methanol:chloroform mixture.

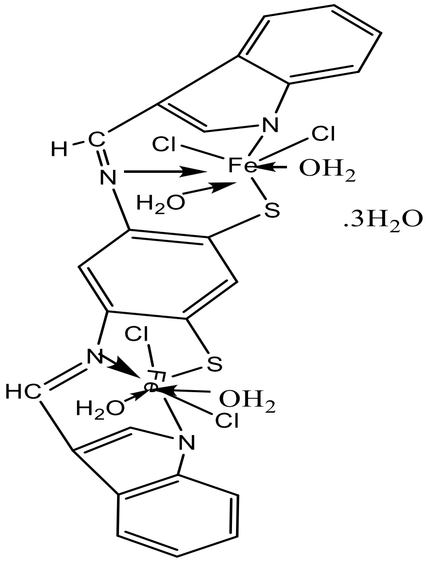

4,6-diaminobenzene-1,3-dithiol

1*H*-indole-3-carbaldehyde

4,6-bis(((*E*)-(1*H*-indol-3-yl)methylene)amino)benzene-1,3-dithiol







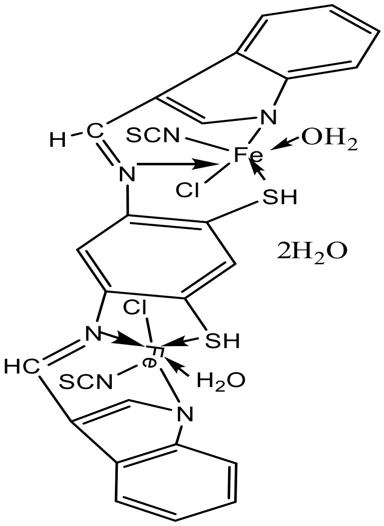


Table 1: Physical characteristics and elemental analyses of the ligand and its complex

Empirical formula	Formula Mwt	Mwt g/ mol	Dec.˚ C	Yield %	%Elemental Analysis Found % (Calculated)				
					C	**H**	**N**	**M**	**Cl**

[MIB]	$C_{18}H_{14}N_6S$	346.10	179	58	62.41 (62.41)	4.07 (4.07)	24.26 (24.26)	-	-
$[Cu_2(MIB)Cl_2].2H_2O$	$Cu_2C_{24}H_{20}N_4O_2S_2Cl_2$	658.56	210	60	43.21 (43.77)	3.11 (3.06)	8.30 (8.51)	19.70 (19.30)	10.68 (10.77)
$[Cu_2(MIB)(NCS)_2].H_2O$	$C_{26}H_{18}Cl_2Cu_2N_6OS_4$	68.80	280	70	45.01 (45.54)	2.52 (2.65)	12.88 (12.25)	19.34 (19.53)	-
$[Fe_2(MIB)Cl_4(H_2O)_2].3H_2O$	$C_{25}H_{32}Cl_4Fe_2N_4O_7S_2$	818.16	270	65	36.84 (36.26)	3.21 (3.94)	13.11(13.65)	15.72 (15.99)	17.82 (17.33)
$[Fe_2(MIB)(NCS)_2(H_2O)_2Cl_2].2H_2O$	$C_{26}H_{22}Cl_2Fe_2N_6O_3S_4$	777.33	250	55	40.10 (40.17)	2.30 (2.85)	10.43 (10.81)	14.20 (14.37)	9.97 (9.12)

Results and discussion

To the best of our knowledge, the ligand was prepared and recrystallized from a chloroform: ethanol mixture, with spectroscopic characterization reported for the first time. NMR spectroscopy at ^{1} H NMR, as well as other techniques, were used to confirm the molecular structure [3]. The structures of the Cu(II) and Fe(III) complexes were also confirmed using various analytical and spectroscopic techniques. Only two of the metal complexes are electrolytic in nature, according to their molar conductance values (measured in 10^{-3} M DMSO solution). Copper (II) iron (III) complexes$[Cu_2(MIB)(NCS)_2].H_2O$are a non-electrolyte.Metal complexes have low solubility in water,chloroform,ethanol, and most organic solvents, but high solubility in DMF and DMSO, and all have high melting points [4].

2.1. Nuclear magnetic resonance spectroscopy

The ligand was studied using ^{1}H NMR spectroscopy. The two amino (NH–) signals observed at 6.65 and 11.73 ppm. Furthermore, the signals of the methylene groups at 3.86 and 7.67 ppm in the ligand appears upfield as a singlets[7]. Furthermore, the multiplateprotons of aromatic groups in range (6.78-8.95) ppm.Also the two signals appear upfield (at 8.93 and 9.80 ppm, respectively), indicating that the Schiff base reaction involves both (CH=N) groups strongly supports the formation of the ligand in proton spectra, respectively. And all the signals observed at 3.84 and 4.96 ppm for the CH= and SH groups,respectively [8].

2.2. IR spectral analysis

Only [Cu_2(MIB)Cl_2].2H_2O and[Fe_2(MIB)Cl_4(H_2O)$_2$].3H_2O have the metal ion coordinated to the deprotonated ligand in SH groups, and[Cu_2(MIB)(NCS)$_2$].H_2O and [Fe_2(MIB)(NCS)$_2$(H_2O)$_2$$Cl_2$].2$H_2O$ are the coordinated to ligand in SH group without deprotonation, according to elemental analysis. The SH stretching frequency was observed as a medium intense band at 2648cm^{-1}, indicating minimal H-bonding. The spectra of [Cu_2(MIB)(NCS)$_2$].H_2O and [Fe_2(MIB)(NCS)$_2$(H_2O)$_2$$Cl_2$].2$H_2O$ complexes show positive shifts of 20-40 cm^{-1} when compared to that of the [Cu_2(MIB)Cl_2].2H_2O and [Fe_2(MIB)Cl_4(H_2O)$_2$].3H_2O,these bands disappeared.The bands observed at 3264–3287 cm^{-1} in the spectra of the metal complexes are broad and suggest the presence of coordinated water molecules may overlap the bonded SH group.The lowering of the stretching frequency of the C=N of the ligand at 1631 cm^{-1} by 23–30 cm^{-1} upon complexation supports the coordination of the N atom of the imine:component of the Schiffbase with the central metal ion, with the greatest reduction observed in [Cu_2(MIB)(NCS)$_2$].H_2O[9]. The bands widened as a result of the complexation, adding to the group's ability to coordinate with metal ions.The observation of the stretching vibration (C-N) at 2127 cm^{-1} for [Cu_2(MIB)(NCS)$_2$].H_2O and 2031cm^{-1} for [Fe_2(MIB)(NCS)$_2$(H_2O)$_2$$Cl_2$].2$H_2O$ predicts thiocyanate and isothiocyanate bonding modes. The observation of the CS in the range 761–790 cm^{-1} strongly supports these conclusions.

Table 2: IRspectra vibrations (cm^{-1}) for the ligand and its complexes

Comp.	ν (O-H)$_{water}$	ν (N-H)	**ν(C-N)** **ν(C-N)**$_{thiocyanate}$	**SH**	**ν(C=N)**	**CS** **CS** $_{thiocyanate}$
[MIB]	-	3129	2112	2621	1631	- 765
[Cu_2(MIB)Cl_2].2H_2O	-	3170	2122 -	-	1604	- 778
[Cu_2(MIB)(NCS)$_2$].H_2O	-	3153	- 2118	2644	1601	- 7 85

$[Fe_2(MIB)Cl_4(H_2O)_2].3H_2O$	3272	3165	2127 -	-	1608	-
$[Fe_2(MIB)(NCS)_2(H_2O)_2Cl_2].2H_2O$	3287	3179	- 2031	2651	1606	796

2.3. Electronic spectra of the ligand and its complexes

The ligand has a maximum absorbance (λmax) of 33,670 and 33,445 cm^{-1} in DMF and DMSO, respectively, which is attributed to the π→π* intra-ligand transition[10]. The band seen at 21,459 cm^{-1} for$[Cu_2(MIB)Cl_2].2H_2O$ complex is thought to be a charge transfer band that obscures the d–d transition in DMF. In DMSO, however, two transitions are visible: the charge transfer band at 20,905 cm^{-1} and the band at 11,612 cm^{-1} indicating that$[Cu_2(MIB)Cl_2].2H_2O$ complex assumed a square planar geometry in DMSO. With the square planar geometry proposed, Complex $[Cu_2(MIB)(NCS)_2].H_2O$ showed similar but lower energies in DMSO compared to DMF in the range 16,103–14,970 cm^{-1}[11].$[Cu_2(MIB)(NCS)_2].H_2O$complex is expected to undergo a ligand to metal charge transfer transition, which is only observed at 22,779 cm^{-1} in DMSO[12].The electronic spectra of Fe(III) complexes are dominated by π→π* and n →π* transitions of the ligand with the most common octahedral geometry, as previously reported. In DMF and DMSO, all of the iron(III) complexes show a medium to intense band in the range 28,923-18,754cm^{-1} and 28,583-19,857cm^{-1}. The charge transfer transition and/or d–d transition peculiar to a Fe^{3+} ion in a 7-coordinate and octahedral configuration, respectively, are assigned to this transition in $[Fe_2(MIB)Cl_4(H_2O)_2].3H_2O$and $[Fe_2(MIB)(NCS)_2(H_2O)_2Cl_2].2H_2O$. Figure 1 illustrates the proposed structures[13].

Table3. Data of electronic spectra of the compounds

Compound	**ʌm. S.cm^2 /mol(DMSO)**	**úcm^{-1} (DMF)**	**úcm^{-1} (DMSO)**	**geometric**
[MIB]	-	34,722 33,423	31,324 30,156	-
$[Cu_2(MIB)Cl_2].2H_2O$	11.65	36,76	32,154,	Square Planar

		5 33,113 21,459	28,653, 20,921, 11,682	
$[Cu_2(MIB)(NCS)_2].H_2O$	17.65	37,175 30,211 16,103	32,680, 22,779, 14,970	Square Planar
$[Fe_2(MIB)Cl_4(H_2O)_2].3H_2O$	9.24	32,787 27,397 18,797	35,461 28,736	7-coordanate
$[Fe_2(MIB)(NCS)_2(H_2O)_2Cl_2].2H_2O$	10.14	32,051 27,322 25,381 21,645	32,051 28,329 21,368	octahedral

2.4. Catechol oxidation activity

The catalytic activities of complexes towards the most common substrate 3,5-di-tert-butylcatechol (3,5-DTBC) were investigated in a DMF solution under aerobic conditions. At 400 nm, there was a gradual appearance of a new band with increasing intensity, as revealed by wavelength scans (Figure 1)[14].The kinetics of oxidation of 3,5-DTBC by complexes were determined using the method of initial rates, with the results shown in Figure 3, and involved monitoring the growth of the quinone band at 399 nm as a function of time[12].The rate constant versus concentration data of the substrate were then analyzed using the Michaelis–Menten approach of enzyme kinetics (Figure 3) to obtain the Lineweaver–Burk plot (double reciprocal) shown in Figure 4, as well as the Michaelis binding constant (K_M) values, maximum velocity (Vmax) and rate constant for substrate dissociation (i.e. turnover rates kcat)[14]. The kinetics data are shown in Table; high kcat values indicate high catalytic efficiency.Catalytic efficiency is in the range of $[Fe_2(MIB)Cl_4(H_2O)_2].3H_2O>[Cu_2(MIB)Cl_2].2H_2O>[Cu_2(MIB)(NCS)_2].H_2O>[Fe_2(MIB)(NCS)_2(H_2O)_2Cl_2].2H_2O$. A7-coordinate geometry is the most catalytically active in$[Fe_2(MIB)Cl_4(H_2O)_2].3H_2O$ complex, while a 4-coordinate geometry is the least catalytically active[15].As previously observed in similar biomimetic studies, the presence of thiocyanate has a negative impact on catecholaseactivity.The values of the turnover rates in the Table are very similar to those previously reported in the literature[16].

Table4. The oxidation of 3,5-DTBC to 3,5-DTBQ mediated by 1–4 in DMF was studied using kinetic parameters.

Compound	Vmax(M min^{-1})	K_M (M)	kcat (h^{-1})
$[Cu_2(MIB)Cl_2].2H_2O$	3.21±0.13) X10^{-7}	(2.23±0.06)X 10^{-3}	10.32 ± 0.12
$[Cu_2(MIB)(NCS)_2].H_2O$	2.98 ±0.22) X10^{-7}	(7.50±0.54)X 10^{-3}	6.54 ± 0.32
$[Fe_2(MIB)Cl_4(H_2O)_2].3H_2O$	4.71 ±0.18) X10^{-7}	(4.87±0.06)X 10^{-3}	13.21± 0.25
$[Fe_2(MIB)(NCS)_2(H_2O)_2Cl_2].2H_2O$	2.11 ± 0.20) X10^{-7}	(3.670±0.06)X 10^{-3}	6.68 ± 0.51

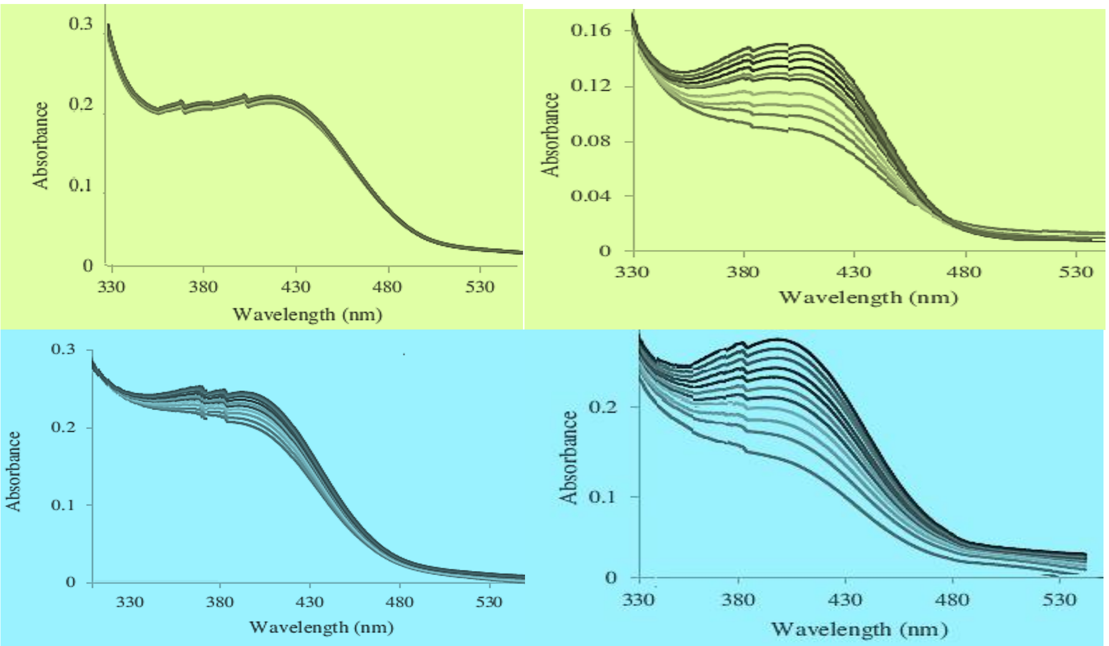


Figure 2: After adding 100 equivalents of 3,5-DTBC to solutions of 1–4 in DMF at 25°C, the quinone band at 399 nm increased. The spectra were taken at 5-minute intervals.

3.4. Catalytic oxidation of 3,5-DTBC

The catecholase activity of the complexes was determined by incubating a 10^{-4} M solution of the complexes in DMF with 100 equiv.3,5-di-tert-butylcatechol (3,5-DTBC) at room temperature under aerobic conditions. Spectral scans were taken every 5 minutes at a regular interval of 5 minutes in the wavelength range 330–550 nm[17]. Because of its low redox potential for the quinone/catechol couple, 3,5-DTBC is the most widely used catechol in catechol oxidase model studies; kinetic experiments (using the method of initial rates) were performed while keeping the concentration of metal complexes constant at $1x10^{-4}$ M and varying the concentration of the substrate (1 $x10^{3}$ M to1 $x10^{2}$ M) in DMF using[18]. The At a wavelength of 399 nm, the formation of 3,5-DTBQ was monitored over time, with each experiment being repeated twice. According to a method previously reported in the literature, the kinetic experiment was followed by the detection of H_2O_2 to provide information on the mechanism of catalytic behavior[19].

2.5. Catecholase activity of complexes: a possible mechanism

1 H NMR spectroscopy was used to identify 3,5-DTBQ [1 H NMR ($CDCl_3$, 300 MHz): 6.22 (d, J = 3.0 Hz, 1H), 6.92 (d, J = 3.2 Hz, 1H)] δ_H = 1.22 (s, 9H),1.31 (s, 9H), 6.41 (d, J = 3.0 Hz, 1H), 6.87 (d, J = 3.2 Hz, 1H)] The fact that H_2O_2 can be measured by iodometric titration indicates that the mechanism is as

follows:When one electron is transferred from catechol to the Cu(II) centre, a semiquinone radical is formed, and the Cu(II) centre is reduced to Cu (I)[20].Dioxygen then binds to the Cu(I) centre, forming Cu(II)-coordinated superoxo species, which quickly absorb electrons from the coordinated semiquinone radical to form coordinated H_2O_2.As a result, the semiquinone is converted to quinone, which dissociates from the metal coordination sphere with H_2O_2 and regenerates the active Cu(II)-catalyst[21]. Some earlier researchers proposed similar mechanisms for DTBC oxidation by some mononuclear complexes that pass through the semiquinoneintermediate[22].

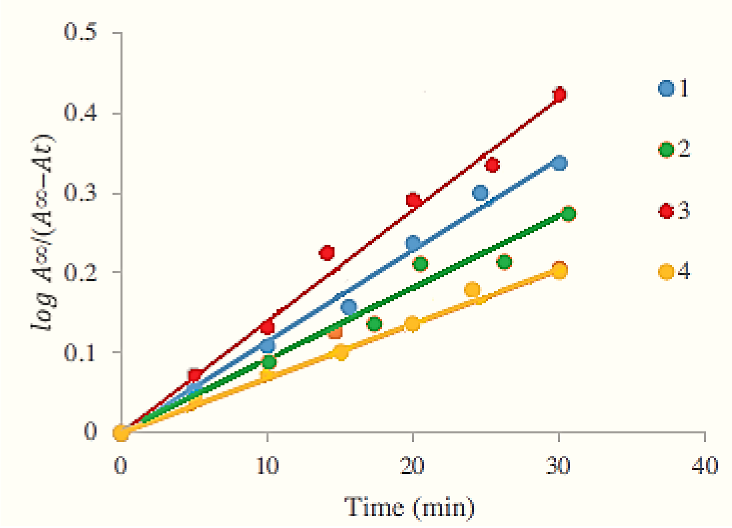
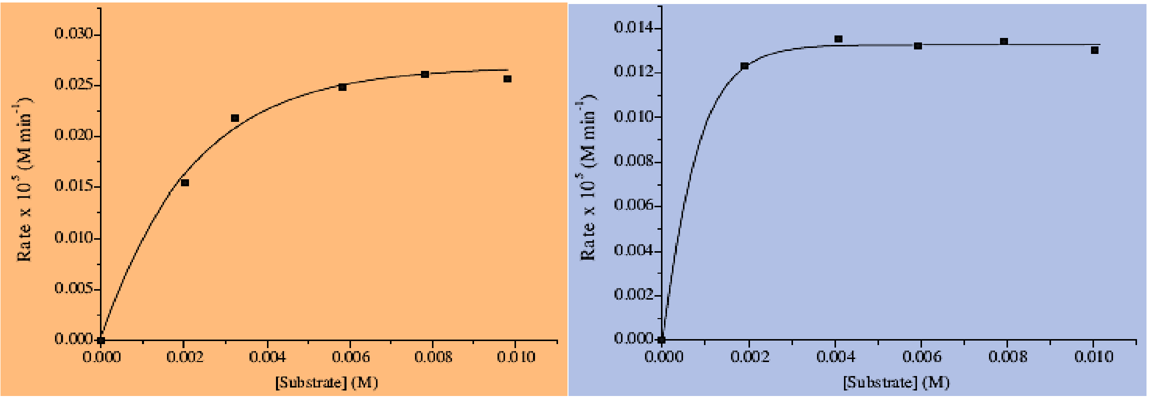


Figure 3: Activity of catecholase in complexes.



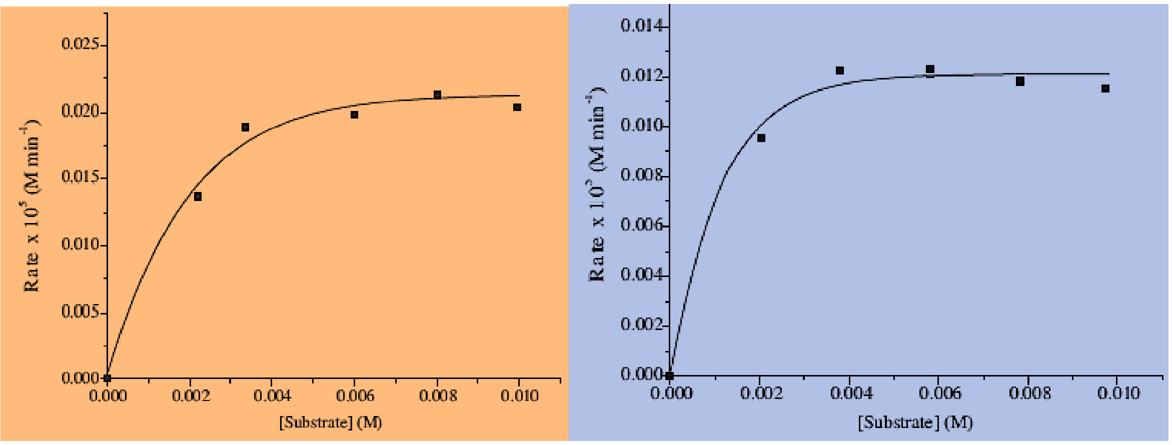


Figure 4. Initial rates of oxidation of 3,5-DTBC catalyzed by complexes plotted against substrate concentration.

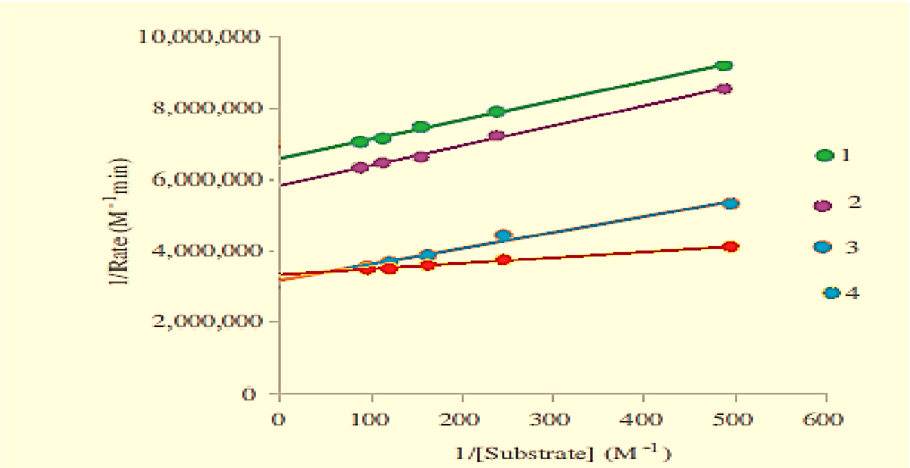


Figure 5. Plots of Lineweaver–Burk for metal complexes.

4. Conclusions

To summarize, we successfully synthesized a new Schiff base, and compared the catecholase activity of its Fe(III) and Cu(II) complexes. The turnover rates of all the metal complexes are moderate. Catecholase activity is reduced when thiocyanate is present within the metal complex. We've also shown that, in the case of[$Cu_2LCl_2.2H_2O$], geometry plays a significant role in the catalytic abilities of metal complexes. The presence of Cu(I) species during the oxidative process is revealed by the identification of H_2O_2 and the isolation of benzoquinone[17].

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