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Manganese 11 Complexes of Salycilhydroxamix Acid with Aniline and Ammonia with Its Properties the Effect on Microorganism

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Abstract:
The salicylichyroxamic acid (SHA) its complex of Mn (II) along with their mixed ligand complexes of ammonia and aniline were prepared in high yields. Both the ligands and complexes were both soluble in methanol, DMSO but are insoluble in chloroform and distilled water. Their melting point were high and falls between 1910C - 400 0C. The IR spectra of the synthesized compound showed the coordination of the ligand through the oxygen of the phenolic NOH and C=O functional groups to the metals. The electronic spectra of the complexes in methanol showed some observable absorption bands of the following transitions; for Mn (SHA) complex transition is at 6A1g(s)→ 4T2g, that of Ammonia mixed complex 6A1g(s)→ 4T2g and that of Aniline mixed complex 6A1g(s)→ 4T2g. All the transitions were in the range of those observed for octahedral complexes. The antimicrobial activity of the ligand SHA (C7H7NO3), the complexes and reference drug were study against two bacterial species staphylococcus auerus and Escherichia coli, and one fungi species candida albicans. This was done using agar disc diffusion method measuring the diameter of inhibitions zone for each compound. The Mn (II) complexes produced in this study showed significant activity against the used micro-organisms. Therefore, they have potential application as anti-microbial agent.

BACKGROUND

Metal complexes of manganese with salicylhydroxamate have been extensively studied due to their potential applications in various fields such as medicine, catalysis, and materials science. Salicylhydroxamic acid (SHA) and its derivatives, including salicylhydroxamates, are known to form stable complexes with transition metals like manganese due to the presence of the hydroxamate functional group which acts as a bidentate ligand. Several research articles have reported the synthesis and characterization of manganese complexes with salicylhydroxamates. Here are a few key references (Chakravarty *et al*., 1986). These metals complexes are often characterized using infrared, UV-Visible, NMR spectroscopy, x-ray crystallograpgraphy and elemental analysis to determine their structures and properties (Mondal *et al*., 2018).

Statement of Research Problem

Microbial infections are a growing problem in contemporary medicine, and the use of antibiotics is common across the world. Antimicrobials are among the most commonly purchased drugs worldwide. They are essential treatments especially in the developing world where infectious diseases are a common cause of death. However, the development of drug resistance as well as the appearance of undesirable side effects of certain antibiotics has led to the search of new antimicrobial agents with the goal to discover new chemical structures which overcome the above disadvantages (Ahmad and Beg, 2001; Poole, 2001).

Justifications of the Study

Salicylhydroxamic acid (SHA) has been reported to possess significant biological activities against a wide range of microbial organisms including bacteria and fungi organisms (Adegoke *et al*., 2019). Therefore, these compounds have potential as antibiotics. SHA is a drug that is a potent and irreversible enzyme inhibitor of the urease enzyme in various bacteria and plants and has been used in the treatment of urinary tract infections (Alhadi *et al., 2012)*. SHA has ability to chelate metal ions which has encouraged its application in the formation of metal ion complexes (He *et al*., 2021) which could help in reducing the menace of antibiotic resistance often observed. Little information is available in the literature about the formation of Mn (II) metal mixed complexes of SHA with aniline and ammonia which can enhance the properties of SHA, this work seeks to explore this possibility.

Aim and Objectives of Study

The aim of this study is to synthesize Mn (II) mixed ligand complexes of salicylhydroxamic acid with aniline and ammonia and determine their antimicrobial activities. To determine the physical/chemical properties of the synthesized mixed ligand complexes, and to also determine the spectroscopic properties of the synthesized complexes using FTIR and UV.

METHODOLOGY

Synthesis of Binary Complex Mn (II) Salicylhydroxamate

Solid Mn (SHAM).$2H_2O$ in 30Ml of MeOH was dissolved in a hot (80°C) aqueous solution (90mL) of SHAM (0.8g). The pH of the resulting solution was raised to 5.5 using 0.1M NaOH solution where upon a copious creamy precipitate was obtained. After standing at room temperature for several hours this was suction filtered, washed with the warm water and dried over silica gel in desiccators.

$$MnSO_4\ .7H_2O + 2(C_7H_7NO_3) \xrightarrow{2NaOH} Mn(C_7H_6NO_3)_2 + Na_2SO_4 + 9H_2O \quad ....1$$

Synthesis of Mixed Complex of Mn (II), SHA and Ammonia

The mixed complex of Mn with SHA and Ammonia (NH_3) was prepared by adding a solution of $MnSO_4\ .7H_2O$ (0.4g) in methanol (30mL) was added to a stirring solution of SHAM (0.8g) in methanol (50mL). NH3 (0.4CM^3) and was stirred vigorously for about 30 minute, and left to stand in refrigerator for several days to form Dark brown crystals The precipitate crystals were filtered, washed several times 50% (v/v) methanol-water to remove any traces of the unreacted starting materials. Finally, the complexes were washed with the diethyl ether and dried in vacuum desiccators over silica gel.

$$MnSO_4.7H_2O + 2(C_7H_7NO_3) + 2NH_3 \xrightarrow{2NaOH} Mn(C_7H_6NO_3\ NH_3)_2 + Na_2SO_4 + 9H_2O. \quad ....2$$

Synthesis of Mixed Complex of Mn (II) and Aniline

The mixed complex of Mn (II) with SHA ($C_7H_7NO_3$) and Aniline(C_6H_7N) was prepared by adding a solution of $MnSO_4\ .7H_2O$ (0.4g) in methanol (30mL) was added to a stirring solution of SHA (0.8g) in methanol (50mL). Aniline(0.2cm^3) and was stirred vigorously for about 30 minute, and left to stand in refrigerator for several days to form dark brown crystals. The precipitate crystals were filtered, washed several times 50% (v/v) methanol-water to remove any traces of the unreacted starting materials. Finally, the complexes were washed with the diethyl ether and dried in a vacuum desiccator over silica gel.

$$MnSO_4 + 2(C_7H_7NO_3) + 2C_6H_7N_3 \xrightarrow{2NaOH} Mn(C_7H_6NO_3\ C_6H_7N_3)_2 + Na_2SO_4 + 2H_2O \qquad \text{........3}$$

RESULTS

Physiochemical Properties

Physiochemical properties of the prepared synthesized compounds were presented Table below.

Table 1: Physico-chemical properties of the synthesized ligand and complexes

s/no	Compound	Molar Mass (g)	Melting/ Dec. point (^{0}C)	Conductivity (μScm^{-1})	Colours	% Yield	% Metal
1	$C_7H_7NO_3$	154.14	170	42.07	Creamy	80	
2	$Mn(C_7H_6NO_3)_2$	359.2	400	12.44	creamy	81	14.71 (15.21)
3	$Mn[(C_7H_6N0_3)(NH_3)]_2SO_4$	489.32	400	108	Dark brown	90	13.97 (10.18)
4	$Mn[(C_7H_6NO_3)(C_6H_5NH_2)]_2$	545.47	145	53.48	Dark brown	56	19.47 (10.02)

Physical Properties of all the Prepared Manganese (II) Complexes

As presented in Table 1, complexes of manganese (II), that is, $Mn(C_7H_6NO_3)_2$, $Mn(C_7H_6NO_3)_2(NH_3)_2SO_4$ and Mn $(C_7H_6NO_3)_2(C_6H_7N\ )_2$ obtained in this study where crystalline in nature. $Mn(C_7H_6NO_3)_2$ was creamy, $Mn(C_7H_6NO_3)_2(NH_3)_2SO_4$ was dark brown and $Mn(C_7H_6NO_3)_2(C_6H_7N\ )_2$ was also dark brown in coloration. The percentage of the complexes prepared were 81%, 90% and 56% for $Mn(C_7H_6NO_3)_2$, $Mn(C_7H_6NO_3)_2(NH_3)_2SO_4$ and Mn $(C_7H_6NO_3)_2(C_6H_7N\ )_2$ respectively (Table 1). All the complexes were quite stable in air and light. The melting points of the complexes were 400°C for $Mn(C_7H_6NO_3)_2$, 400°C for $Mn(C_7H_6NO_3)_2(NH_3)_2SO_4$, and 145°C for $Mn(C_7H_7NO_3)_2(C_6H_7N\ )_2$ as presented in Table 1. The sharp melting point observed is an indication of the absent of impurity (or minimal) in the prepared ligand and the high melting may signifies the polymeric nature of the complexes as reported by Aliyu and Nwabueze (2009). Also reported in Table 4.1 were the conductivities values of all the complexes. Complex $Mn(C_7H_6NO_3)_2$ had conductivity value of 12.44 μS cm^{-1} and complex $Mn(C_7H_6NO_3)_2(C_6H_7N\ )_2$ had conductivity value 53.48 μS cm^{-1}. These values were considered low and indicated virtually non-electrolytes nature of the complexes, On the other hand, complex $Mn(C_7H_6NO_3)_2(NH_3)_2SO_4$ showed a very high conductivity value 193.2 μS cm^{-1} (see Table 1) indicating an electrolyte nature of this complex(Roy *et al.*, 2007).

Solubility of the Prepared Complexes

The result of the solubility studies of the synthesized ligand and complexes were presented in Table below.

Table 2: Solubility profile of synthesized ligand and complexes

Complexes	Methanol	Water	Chloroform	DMSO
$C_7H_7NO_3$	S	IS	IS	S
$Mn(C_7H_6NO_3)_2$	S	IS	IS	S
$Mn[(C_7H_6N0_3)(NH_3)]_2 SO_4$	S	IS	IS	S

$Mn[(C_7H_6NO_3)\ (C_6H_5NH_2)]_2$	S	IS	S	S

KEY: S – Soluble, IS – insoluble, PS – partially soluble.

Solubility of all the Prepared Manganese (II) Complexes

Solubility profiles of manganese (II) complexes were represented in Table 2. As presented in the table, complexes $Mn(C_7H_6NO_3)_2$ and $Mn(C_7H_6NO_3)_2(NH_3)_2SO_4$ were soluble in both DMSO and methanol but insoluble in chloroform and water. Prepared complex $Mn(C_7H_6NO_3)_2(C_6H_7N)_2$ was soluble in DMSO, methanol and chloroform but insoluble in water. This may be due to polymeric nature of the complexes (Abramov, 2015).

FTIR Spectra of Ligand and Synthesized Compounds

The IR spectra of the prepared compound are provided in appendix, while the identified diagnostic bands are given in Table below.

FTIR charts diagnostic band of the compounds (cm-^{1}) complexes

Compound	√(OH)	√(C=O)	D√(C=O)	√(C-N)	D√(C-N)	√(N-O)	D√(N-O)	√(M-O)	√(M-N)
$C_7H_7NO_3$	3489	1617	-----	1353	-----	906	-----	-----	-----
$Mn(C_7H_6NO_3)_2$	3426	1594	23	1386	33	920	14	608	546
$Mn[(C_7H_6N0_3)\ (NH_3)]_2\ SO_4$	3462	1582	35	1350	03	908	02	612	572
$Mn[(C_7H_6NO_3)\ (C_6H_5NH_2)]_2$	3446	1576	41	1399	46	988	82	658	603

FTIR Spectra of All the Prepared Manganese (II) Complexes

Significant absorption band observed for the synthetized Mn (II) complexes were also presented in Table 3. $Mn(C_7H_7NO_3)_2$ complex showed significant band at 3428 cm^{-1}, $Mn(C_7H_6NO_3)_2(NH_3)_2SO_4$ complex showed significant band at 3493 cm^{-1}, and $Mn(C_7H_7NO_3)_2(C_6H_7N)_2$ complex showed significant band at show band 3446 cm^{-1}. This band were assigned to √(OH) stretching frequency similar to that of the ligand SHA. However, √(OH) for $Mn(C_7H_7NO_3)_2$ shifted to a lower value by 61 when compared to that of SHA, that of $Mn(C_7H_7NO_3)_2(C_6H_7N)_2$ shifted to a lower value by 43 when compared to that of SHA (see Table 3). This showed coordination through the OH group of the ligand. The complexes $Mn(C_7H_7NO_3)_2$, $Mn(C_7H_6NO_3)_2(NH_3)_2SO_4$ and $Mn(C_7H_7NO_3)_2(C_6H_7N)_2$ also shows band at 1617,1599 and 1576 cm^{-1} designated as √(C=O) stretching frequency. A shift were observed for $Mn(C_7H_6NO_3)_2(NH_3)_2SO_4$ and $Mn(C_7H_7NO_3)_2(C_6H_7N)_2$ √(C=O) stretching frequency when compared to that of ligand SHA to a lower significant value of 18 and 41 cm^{-1} respectively indicating a coordination at this bands √(C=O) (Shankar et, al 2014). The complexes $Mn(C_7H_7NO_3)_2$, $Mn(C_7H_6NO_3)_2(NH_3)_2SO_4$ and $Mn(C_7H_7NO_3)_2(C_6H_7N)_2$ also shows band at 1387, 1395 and1399 cm^{-1} for √(C-N) which shifted to a higher significant value of 34, 42 and 46 cm^{-1} respectively from that of ligand SHA indicating a coordination at this bands √(C-N). Complexes $Mn(C_7H_7NO_3)_2$, $Mn(C_7H_6NO_3)_2(NH_3)_2SO_4$ and $Mn(C_7H_7NO_3)_2(C_6H_7N)$ also shows band at 922, 921 and 988 cm^{-1} assigned to √(N-O) frequency and which shifted from that of ligand SHA to a higher significant value of 16, 15 and 82 respectively indicating a coordination at this bands, they also showed new bands at 517, 617 and 685 cm^{-1} assigned to √ (M- O) showing metal- oxygen coordination. Also bands at 56,592 and 603 cm^{-1} assigned √(M-N) indicating a metal- nitrogen coordination were also observed (Shotor *et al.*, 2010).

UV/visible Electronic Spectra of Synthesized Compounds

The UV-visible of the ligands and prepared metal complexes where done in methanol and are shown in Appendix, while the max of their transitions are mention in the table below.

Electronic spectral data (in nm) of all the synthesize Ligand and complexes

Compounds	λ_{max} nm (cm^{-1})	Transitions
$C_7H_7NO_3$	224(44642)	$n \rightarrow \prod^*$
$Mn(C_7H_6NO_3)_2$	475(21052)	$^6A_{1g(s)} \rightarrow {}^4T_{2g}$
$Mn[(C_7H_6N0_3)\ (NH_3)]_2\ SO_4$	520(19230)	$^6A_{1g(s)} \rightarrow {}^4T_{2g(D)}$
$Mn[(C_7H_6NO_3)\ (C_6H_5NH_2)]_2$	480(21052)	$^6A_{1g(s)} \rightarrow {}^4T_{2g(D)}$

UV/visible Electronic Spectra of Synthesized Compounds

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$Mn[(C_7H_6NO_3)\ (C_6H_5NH_2)]_2$	480(21052)	$^6A_{1g(s)} \rightarrow {}^4T_{2g(D)}$

Electronic Spectra of the Prepared Manganese (II) Complexes

The electronic spectra of the UV-visible of the complexes were presented in Table 4. Complexes $Mn(C_7H_6NO_3)_2$, $Mn(C_7H_6NO_3)_2(NH_3)_2SO_4$ and $Mn(C_7H_6NO_3)_2(C_6H_7N)_2$ show band at 475nm, 520nm and 480nm respectively. Manganes have electronic configuration of d^5 and a spectroscopic ground term symbol of ^6S. S-orbital here is non-degenerate and cannot be split by either octahedral or a tetrahedral. The field the intensities are much weaker than for spin-allowed d-d spectra (Adediji *et al.*, 2013). Octahedral or tetrahedral structure of the complexes was therefore suspected.

Antimicrobial Activity

Antimicrobial activities of SHA, and its complexes and the reference drugs were given in the Table

Table 3: Antimicrobial activities of SHA and its complexes

S/no	**Compound**	***Staphylocccocus aureus* DIZ (mm)**		***Escherichia coli* DIZ (mm)**		***Candida albicans* DIZ (mm)**	
1	$C_7H_7NO_3$	10.0	+	13.0	+	9.0	+
2	$Mn(C_7H_6NO_3)_2$	18.0	++	13.0	+	14.0	+
3	Positive control	18.0	+++	16.0	++	22.0	+++
4	Negative control	NSI	NSI	NSI	NSI	NSI	NSI

Note: DMSO (-ve control); chloramphenicol (bacteria +ve control); Nystatin (fungi +ve control), DIZ (diameter of zone of inhibition); NSI (no significant inhibition); ++ = moderate activity (DIZ: 9 – 14 mm), +++ = good activity (DIZ:15 – 18 mm); ++++ = very good activity (DIZ:19 – 22 mm), +++++ = significant activity (DIZ: 23 – 26m).

Antimicrobial Screening of all the Prepared Manganese (II) Complexes

The antimicrobial activities of Mn (II) complexes were presented in Table 5. As reported in the table, complex of $Mn(C_7H_6NO_3)_2$ showed DIZ values of 18.0, 14.0 and 13.0 respectively which as designated as moderate activity towards *Staphyloccocus auerus* and week inhibition towards *Candida albicans* and *Escherichia coli*. $Mn(C_7H_6NO_3)_2(C_6H_7N)_2$ had DIZ values of 26.0, 16.0 and 21.0 and were said to be very good inhibition towards *Staphylocccocus auerus* and *Escherichia coli* and good activity towards *Candida albicans* respectively as proposed by Fazasy (2014). The synthesized complex $Mn(C_7H_6NO_3)_2(NH_3)_2SO_4$ had DIZ values of 22.0, 23.0 and 13.0 and was designated as very good inhibition towards *Staphylocccocus auerus* and *Candida albicans* with week inhibition towards *Escherichia coli*. Similar conclusions were made by Aliyu and Nwabueze (2009).

CONCLUSION

The primary ligand in this study (salicylhdroxamic acid) was prepared by reacting methyl salicylate with hydroxyl amine. This was used to prepare binary complexes via its reaction with manganese (II) tetraoxosulphate (VI) in methanoic medium. Two mixed ligand (ternary) complexes were also prepared using ammonia and aniline as the secondary ligand. The complexes where characterized using FTIR, UV-Vis, solubility, melting point and conductivity. The result showed that most of the compounds were non-electrolytic in nature owing to the low value of their molar conductivity in DMSO except for $[Mn(C_7H_6NO_3)_2(NH_3)_2]SO_4$ which shows high conductivity of 198.08 $\mu S\ cm^{-1}$ and 108.8 $\mu S\ cm^{-1}$. The complexes were found to be soluble in methanol and DMSO and insoluble in water. High melting point were observed for the complexes with $[Mn(C_7H_7NO_3)_2(C_6H_7N)_2]$ having the lowest melting point of 145 ^{o}C. Their solubility pattern and high melting point temperature suggested that they may be polymeric complexes has suggested by AL-Noor (2021), Aliyu and Nwabueze, 2009.

FTIR and UV/Visible result suggested that in the metal-primary ligand complexes, the ligand coordinate with the metal ions via its carbonyl oxygen and the deprotonated OH oxygen atom suggesting a {O, O} coordination. The mixed ligand complexes showed {O, N} coordination, through the carbonyl oxygen of the primary ligand and nitrogen atoms from the secondary ligands. On the basis of this study tetrahedral and octahedral geometry were proposed for the synthesized complexes. Biological screening of the synthesized complexes revealed that they had remarkable antimicrobial activity against *Staphylocccocus auerus*, *Escherichia coli* and *Candida albican.* The mode of action may involve the formation of a hydrogen bond through the nitrogen and oxygen atoms present in the ligands structure active centers of the cell constituents, resulting in interference with the normal cell process as suggested by (Henry *et al.*, 1998). It may also be as a result of electrical attraction between the complexes metal ions and the active centers of the cell. Therefore, complexes synthesized in this study have great potential as antimicrobial agent and a candidate for solving the menace of antibiotics resistance.

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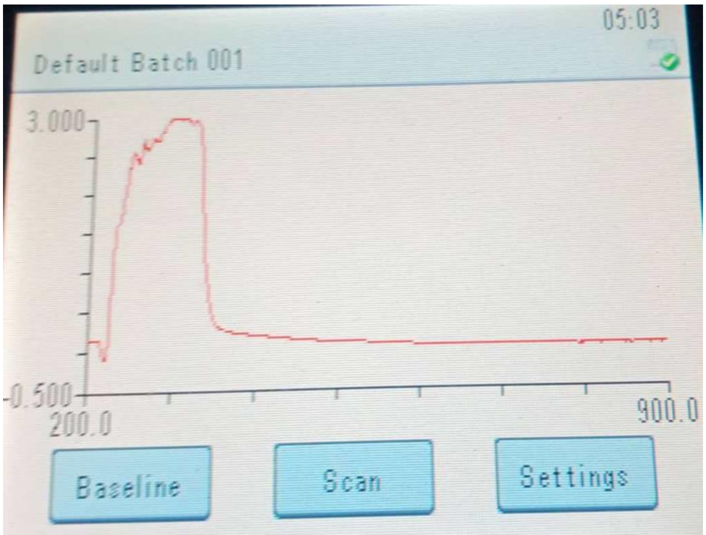
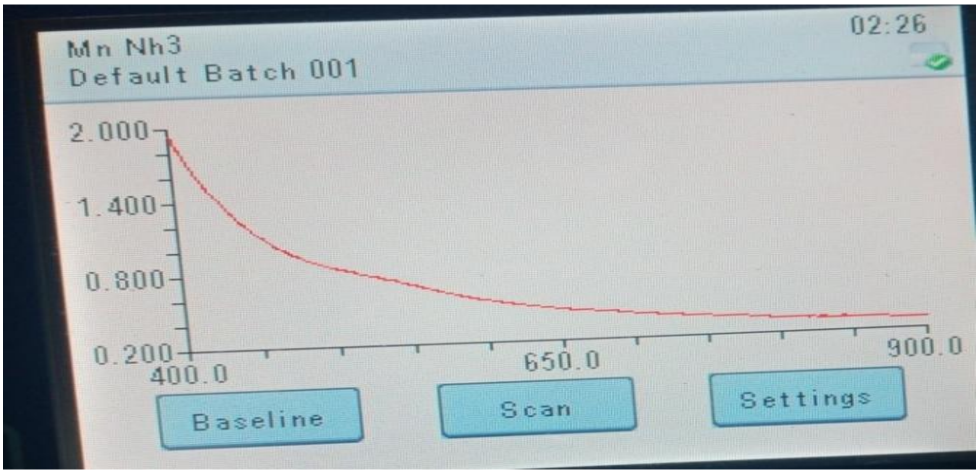
APPENDICES

Appendix Antimicrobial Activities

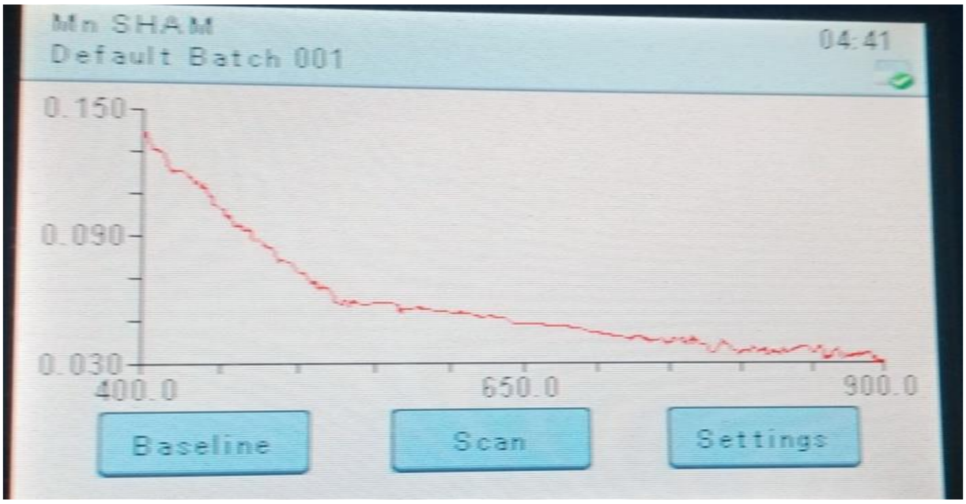
	Staphylococus aureus					***Eschericia coli***					***Candida albicans***				
SAMPLE	Diameter of ZI (mm) Cont. X_1 X_2 X_3 -ve +ve					Diameter of ZI (mm) X_1 X_2 X_3			Cont. -ve +ve		Diameter of ZI (mm) X_1 X_2 X_3			Cont. -ve	+ve
Mn + Sham +NH_3 0.4g	22.0	22.0	22.0	NI	18.5	13.0	13.0	13.0	NI	16.0	23.0	23.0	23.0	NI	22.0
SHAM +Mn+ NH_3 0.4g	18.0	18.0	18.0	NI	18.5	13.0	13.0	13.0	NI	16.0	14.0	14.0	14.0	NI	22.0
Aniline Mn SHAM 0.4g	26.0	26.0	26.0	NI	18.5	21.0	21.0	21.0	NI	16.0	21.0	21.0	16.0	NI	22.0



UV visible spectrum pictures of SHA



UV visible spectrum of, $MN(C_7H_6NO_3)_2(NH_3)_2SO_4$



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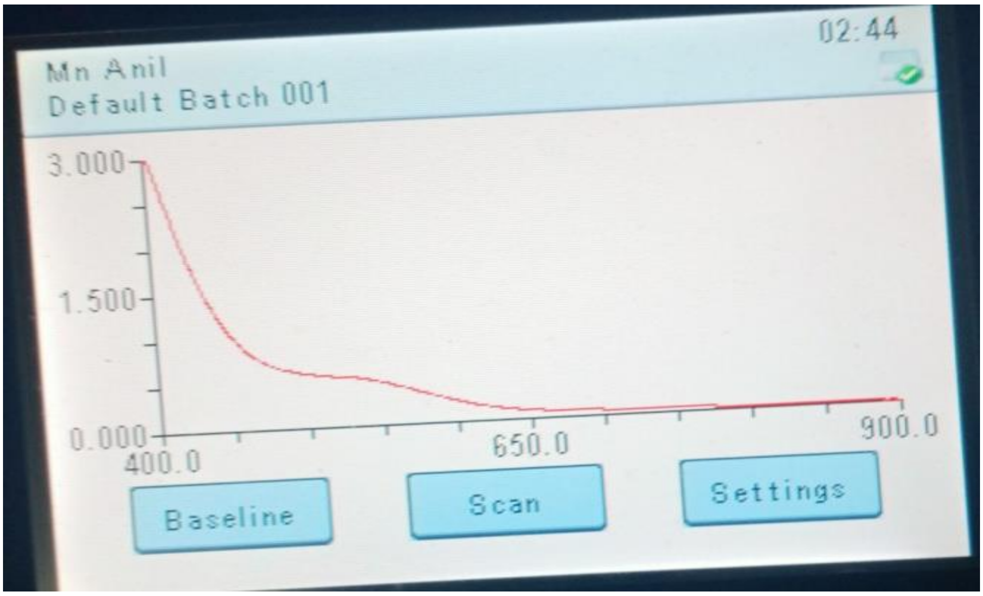
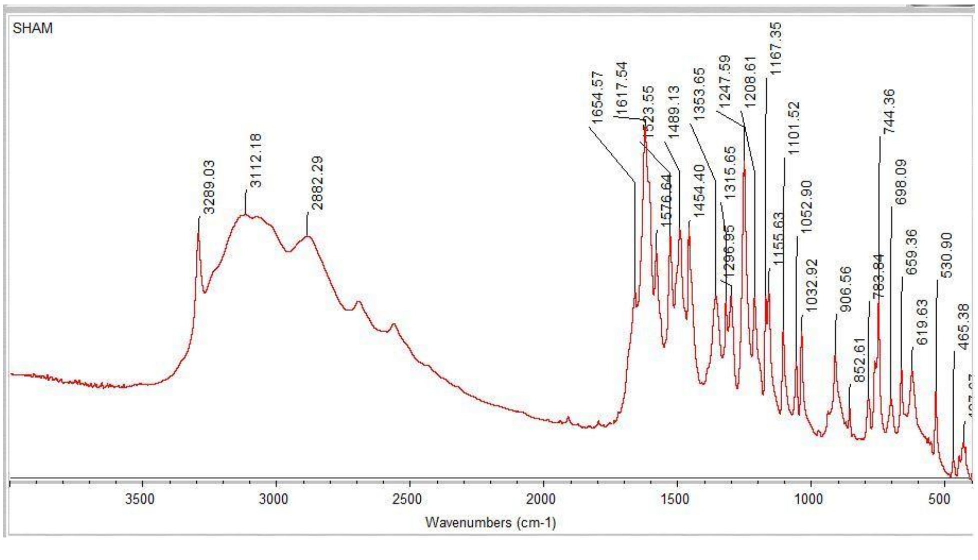
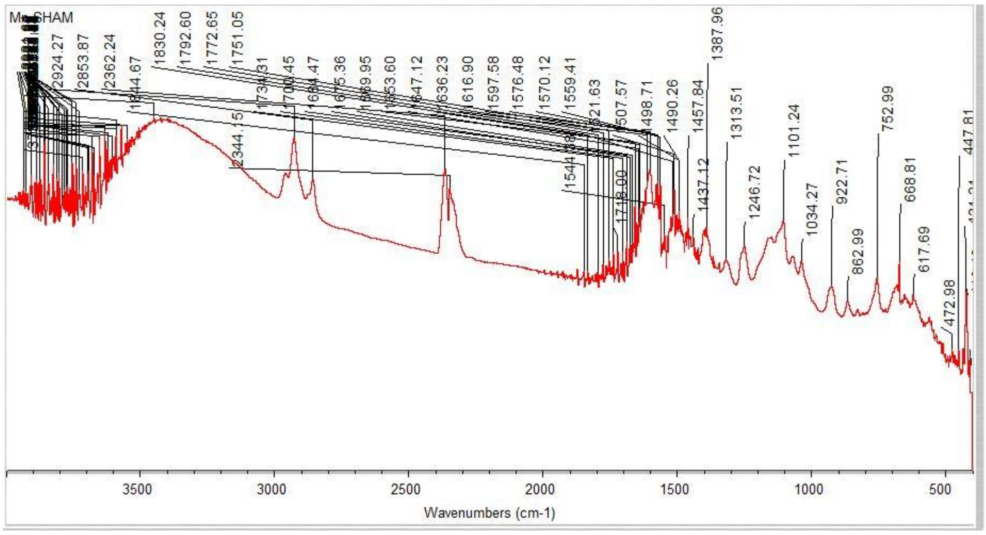
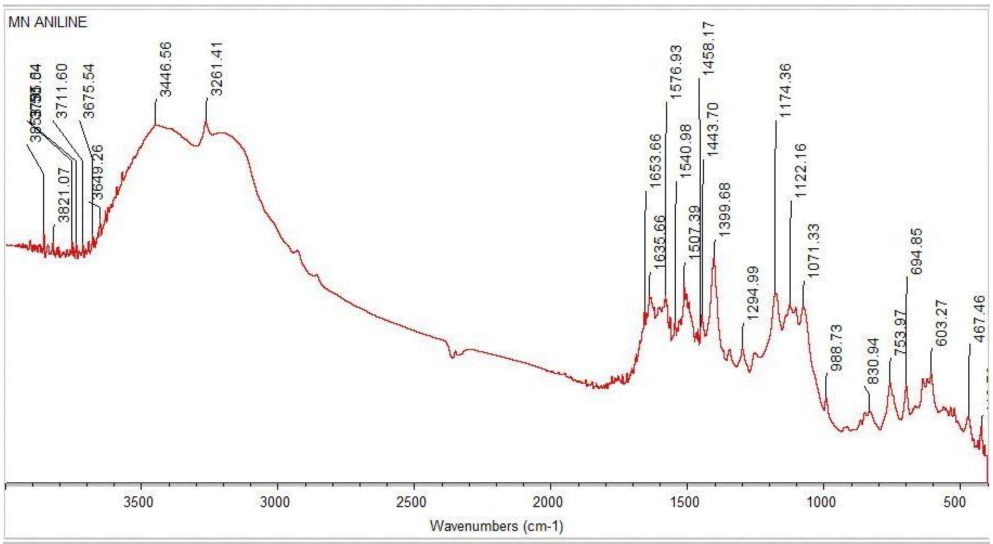


UV visible spectrum of $Mn(C_7H_7NO_3)_2(C_6H_7N\ )_2$

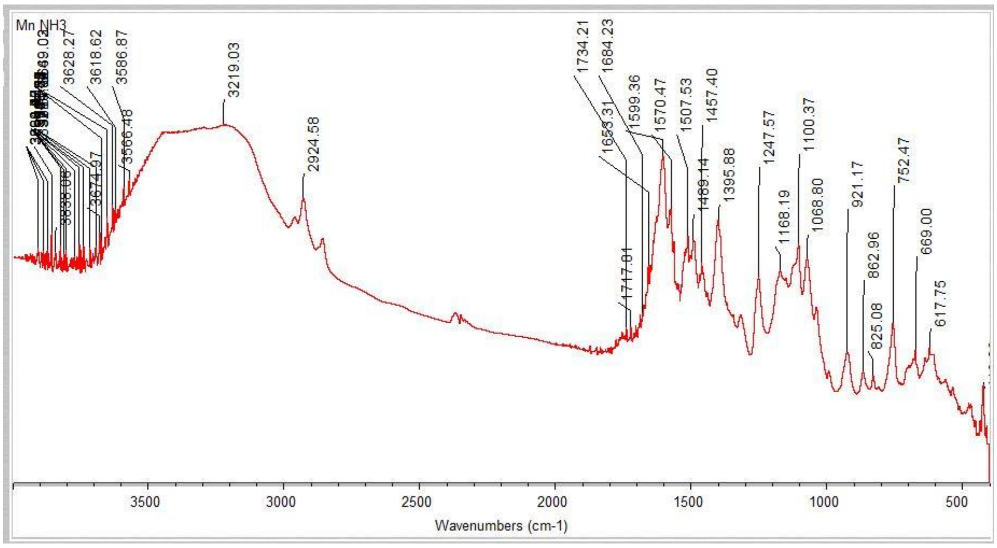




FTIR Spectrum for SHA



FTIR Spectrum for $Mn(C_7H_6NO_3)_2(C_6H_7N)_2$ complex.



FTIR Spectrum for $Mn(C_7H_6NO_3NH_3)_2SO_4$ complex