Journal of Natural Sciences Research www.iiste.org
ISSN 2224-3186 (Paper) ISSN 2225-0921 (Online)
Vol.5, No.5, 2015

Synthesis, Charactrization and Antioxidant Activity of som New Schiff Bases Derived from 2-Hydroxy-1-Naphthaldehyde

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Abstract
Four series of some new Schiff bases formed from reaction of 2-hydroxy-1-naphthaldehyde with 2-hydroxy benzaldhyde and diamino propane have been prepared and characterized. The structure of the synnthesised compounds was confirmed by elemental analysis (CHN), UV-visible, infrared and ^{1}H-NMR techniques. The antioxidant activity of the prepared compounds was studied using scavenger technique. The results indicate that the new Schiff base compounds are very effective as rdical scavengers compared with standarad ascorbic acid (ASC).
Keywords: Schiff bases, Synthesis, Antioxidant activity

1.Introduction
Recently there has been a considerable interest in synthesis of new compounds of Schiff bases due to their biological applications such as antibacterial [Karthikeyan MS. et al.2006, Asiri A.M. and Khan S.A,2010, Patel N.B. and Patel J.C.2011, Maladi S.2013] antifungal [Gune Z. et al.2007, Al-Amiery A.A.et.al.2012] antitumer[Li X.et al.2012, Bahat M.A.et al.2015] and antioxidant [Cheng LX.et al. 2010, Kumar P.P. and Rani B.L. 2011].

The Antioxidant agents such as Shciff bases are very important class of compounds to keep human health due to their ability to scavenge harmful free radicals and therfore preventing some dangerous diseases.These observations encourge us to synthesize some new Schiff baseses and to investigate their structure and antioxidant activities.

2.Experimental
2.1 Materials
1,3 Diamino propane, 2-hydroxy-1-naphthaldehyde and 3-ethoxy-2-hydroxy benzaldhyde were obtained from Aldrich in pure state. 2-Amino benzylamine was obtained from Fluka in pure state. Methanol, ethanol, chloroform and dimethyl sulfoxide were obtained from G.C.C. company and used after being purified according to the standard method cited in the literatures.

2.2 Instruments
CHN elemental analysis was carried out on Thermofingigan flash. Infra-red spectra were recorded by Shimadzu (Japan) FTIR affinity spectrometer as KBR disc in wave number range 4000-250 cm^{-1}.Ultraviolet-visible spectra were recorded by PG(T60UV)-Germany spectrometer. The ^{1}H NMR spectra were recorded by Bruker DRX system AL500 (500MHz) using $CDCl_3$ as a solvent and TMS as a reference. Mas spectra were recorded using work mass selective detector 5973 with 70 ev energy.

2.3 Synthesis of New Schiff Bases
2.3.1 Synthesis of N-(naphthalidene)-N'-(3-ethoxysalicylidine) 1,3- Diamino propane(L_1).
L_1 was prepare by adding (0.01 mole, 1.72 gm) of 2-hydroxy 1-naphthaldhyde in 15 ml of methanol to (0.01 mole, 1.66 gm) of 3-ethoxy-2- hydroxy benzaldhyde in 15 ml of ethanol. The two compounds were mixed and acidified by some drops of glacial acetic acid. (0.01 mole , 0.74 gm) of 1,3-diamino propane was added to the above mixture and then refluxed for 40 minutes.The progress of the reaction was tested by thin layer chromotography (TLC) and when the reaction was completed the solid product was collected by filteration, dried and recrystalized from absolute ethanol.The yield of orange product, m.p. 169-171 ^{0}C , was 85% .m/z 376, 212, 170, 143, 75 (the peak at 376 originates from an L_1 molecular ion $C_{23}H_{24}N_2O_3^+$).
2.3.2 Synthesis of N-(naphthalidene)-N'-(3-ethoxysalicylidene) ortho amino benzyl amine (L_2).
L_2 was similarly prepared from reaction of 2-hydroxy-1-naphthaldhyde and 3-ethoxy-2-hydroxy benzaldhyde mixture with 2-amino benzyl amine.The yield of pale yellow product, m.p. 198-200 ^{O}C, was 86%. m/z 424, 254, 170, 137,75 (tha peak at 424 originates from an L_2 molecular ion $C_{27}H_{24}N_2O_3^+$).
2.3.3 Synthesis of N,N,-di(naphthalidene)-ortho amino benzyl amine (L3).
L3 was prepared by the same procedure from reaction of 2-hydroxy 1-naphthaldhyde and 2-amino benzyl amine.The yield of yellow product, m.p. 223-2250C, was 81%. m/z 430, 260, 143, 126, 90, 77 (the peak at 430

originates from an L3 molecular ion $C_{29}H_{22}N_2O_2$+).

2.3.4 Sythesis of N,N'-di(3-ethoxysalicylidene)-ortho amino benzyl amine (L4).

L4 was prepared by the same procedure from reaction of 3-ethoxy-2-hydroxy benzaldhyde and 2-amino-benzyl amine. The yield of pale orange product, m.p. 214-216 ^{0}C, was 90% . m/z 418, 254, 164, 137, 90, 76 (the peak at 418 originates from an L_4 molecular ion $C_{25}H_{26}N_2O_4$ $^+$).

Scheme(1) shows the synthesis routes for L_1, L_2, L_3 and L_4 Schiff bases.

3.Results and Discussion

3.1 Elemental analysis

Elemental analysis of the synthesized Schiff bases L_1-L_4 is sumerized in Table (1). This indicates that the founded percentages of CHN elements are in good agreement with the calculated values, which means that the synthesis of Schiff bases is succesful.

3.2 Infra-red spectra

The infra-red spectra of Schiff-bases compounds L_1-L_4 are represented in Figures 1, 2, 3 and 4 respectively. The appearance of - C=N stretching vibration for all prepared compounds of Schiff bases at 1610-1624 cm^{-1} indicates that the reaction between diamine derivatives and naphthaldhyde derivatives is successful. Figures 1-4 also indicate the presence of stretching frequencies of OH , aromatic C-H, aliphatic C-H and aromatic C =C bonds. Table(2) summarized streching frequencies of all Schiff base compounds.

3.3 proton NMR Spectra

The proton NMR Spectra of prepared Schiff bases L_1-L_4 are represented in Figures 5, 6, 7,and 8, respectively. These Figures show that the proton of (CH = N) absorbs at (8.93-8.96) which confirms that the synthesis of Schiff bases is succesful. The Figures also show the appearance of resonance of middle and lateral methylene, aromatic C-H, ethoxy groups.

3.4 Ultra-Violet Spectra

All ultra-violet spectra of prepared Schiff bases were characterized by three electronic transitions, the first one composes of one intense band at 334 nm which attributed to $n \rightarrow \pi^*$ electronic transition. The second transition composes of two intense bands at 297 and 225 nm which attributed to $\pi \rightarrow \pi^*$ transitions.

3.5Antioxidant Activity

The Schiff bases L_1-L_4 were subjected to their possible antioxidant activity using stable free radical 2,2-diphenyl-2-picrylhydrazyl (DPPH) as hydrogen accepter. The DPPH radical scavenging activities of tested L_1-L_4 were evaluated after mixing of Schiff bases with DPPH and then incubated in the dark for 30 min and 1 hr. The DPPH radical scavenging activity wih ascobic acid was also assayed for comparsion.The percentage of antioxidant activity was evaluated using the following equation (Al-Amiery A.A.et al. 2012)

$$\text{Antioxidant activity}(\%) = A_0 - A_1 / A_o \times 100$$

Where A_o is the absorbance of the control reaction and A_1 is the absorbance in the presence of the sample or standared ascorbic acid (ASC).DPPH radicals absorb visible light at 517 nm and when antioxidant compounds were added to them the absorption of DPPH decreases. Figures(9) and(10) show the antioxidant activity of Schiff bases L_1-L_4 together with ascorbic acid as standard refrence. As shown in the figures we can see that the Schiff base compounds show high antioxidant properties compared with standard ascorbic acid. This may be attributed to the efficiency of L_1- L_4 to promote hydrogen atom from azomethine and OH groups.

4.Conclusion

The present study demonstrated that the new prepared Schiff bases exhibited feasible antioxidant activity.

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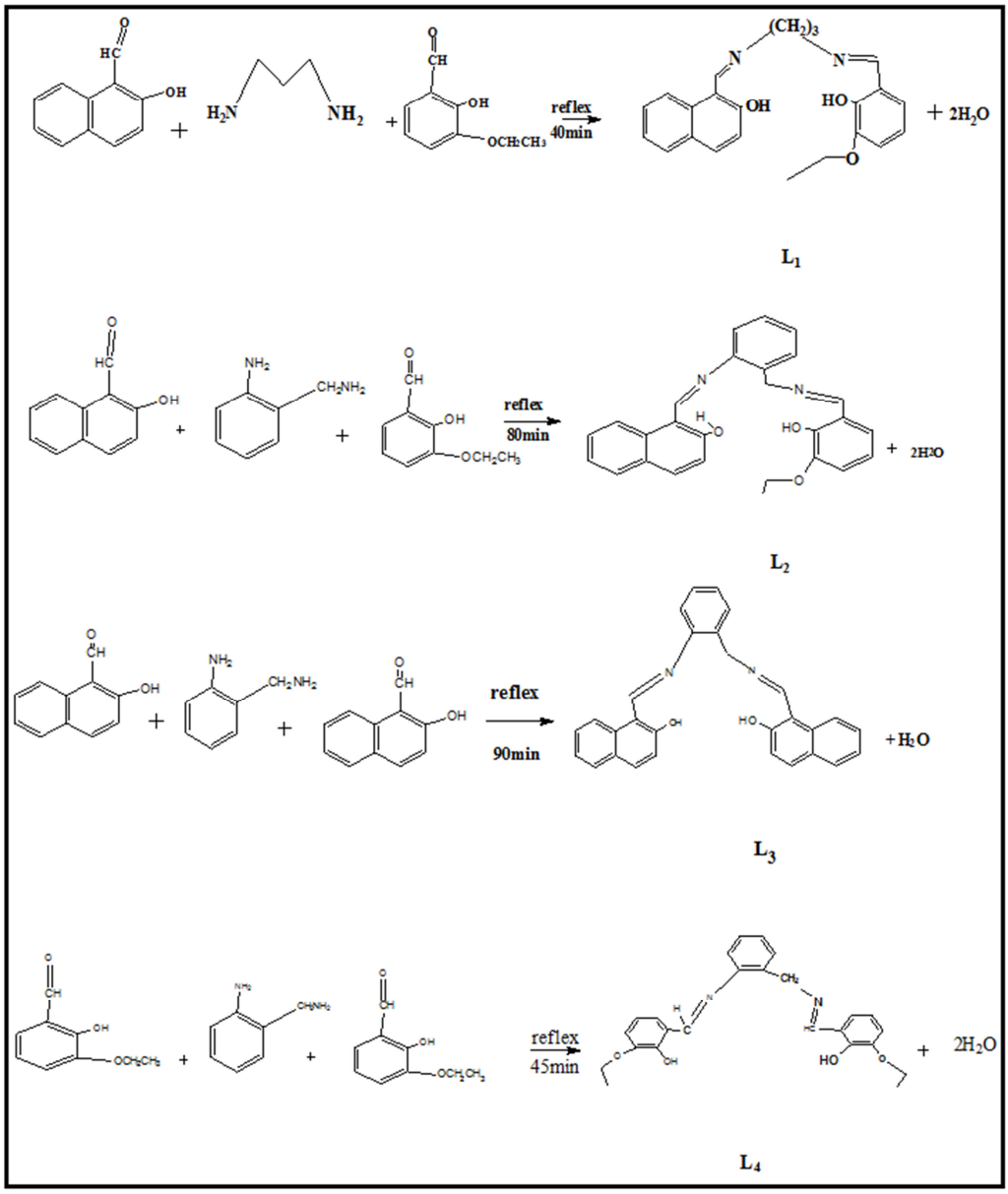


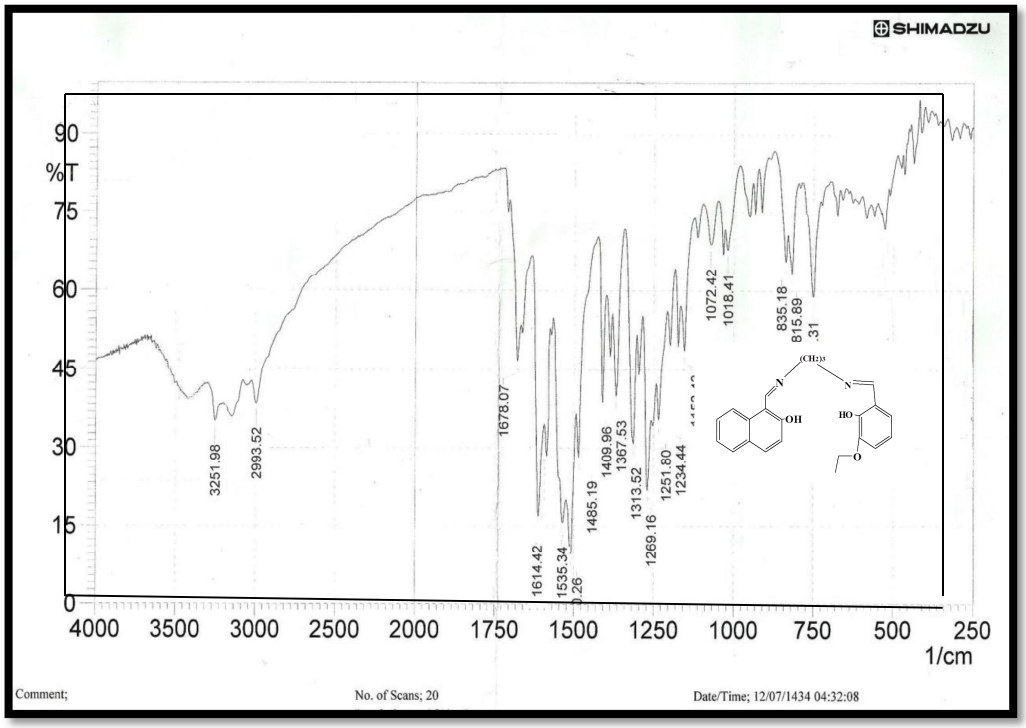
Scheme (1) Synthetic routes of Schiff bases (L_1-L_4)

Table (1) CHN analysis of Schiff bases (L_1-L_4)

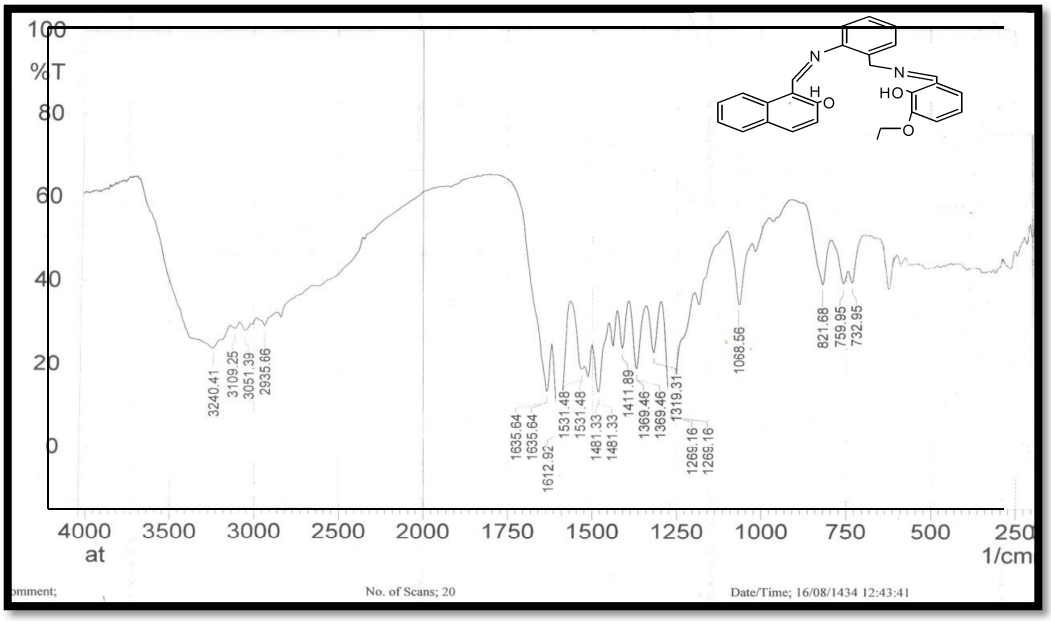
NO	Formula	M.Wt	C%		H%		N%	
			Calculated	Found	Calculated	Found	Calculated	Found
L_1	$C_{23}H_{24}N_2O_3$	376	67.12	67.02	7.4	7.31	7.87	7.82
L_2	$C_{27}H_{24}N_2O_3$	424	76.04	76.03	6.18	6.14	6.57	6.6
L_3	$C_{29}H_{22}N_2O_2$	430	80.22	80.16	6.07	6.03	6.47	6.45
L_4	$C_{25}H_{26}N_2O_4$	418	71.46	71.41	6.83	6.71	6.75	6.66

Table (2) The most important vibration frequences of functional groups in Schiff bases (L_1-L_4)

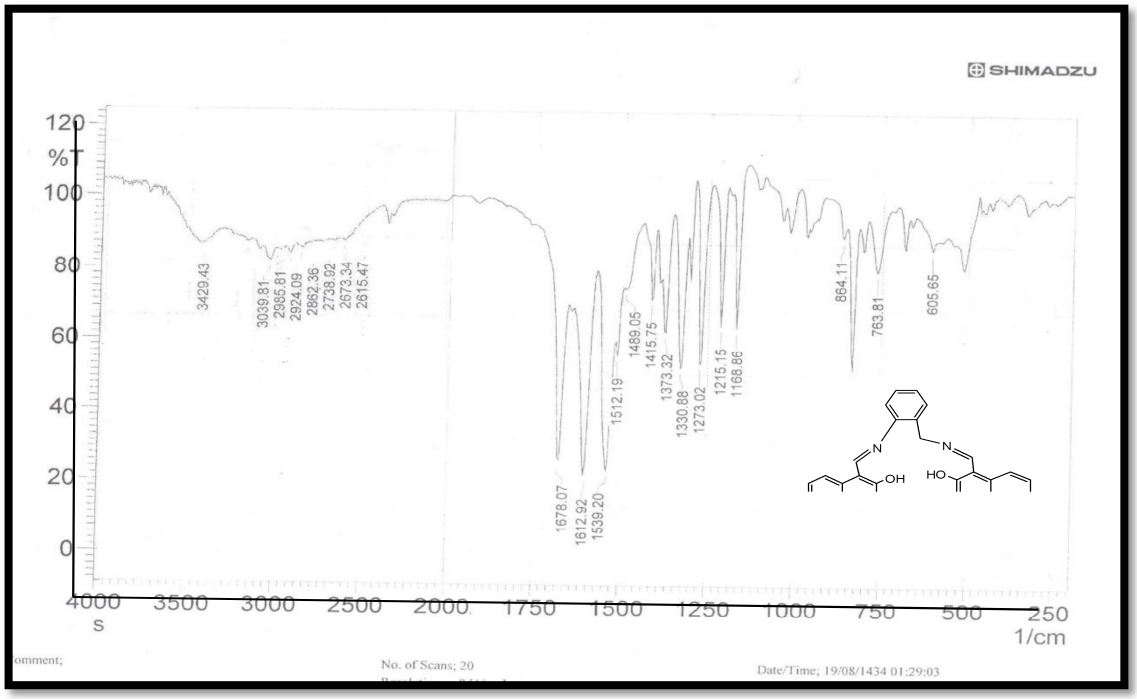
NO	Compound	O-H	C-H (Ar.)	C-H Aliph	C=N	C=O	C-O	C=C
L_1	$C_{23}H_{24}N_2O3$	3430	3062	2943	1614	1678	1269	1510
L_2	$C_{27}H_{24}N_2O_3$	3392	3051	2935	1612	1635	1269	1539
L_3	$C_{29}H_{22}N_2O_2$	3429	3039	2985	1612	1678	1215	1539
L_4	$C_{25}H_{26}N_2O_4$	3125	3034	2970	1616	1631	1257	1570



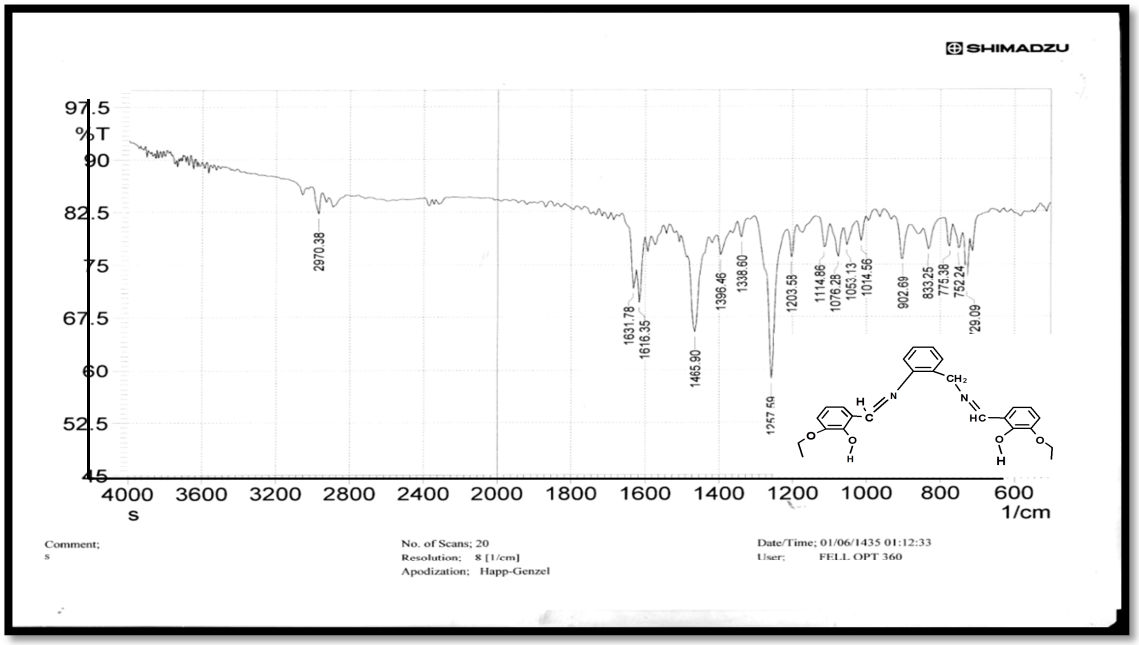
Fig(1) IR spectrum of Schiff base(L_1)



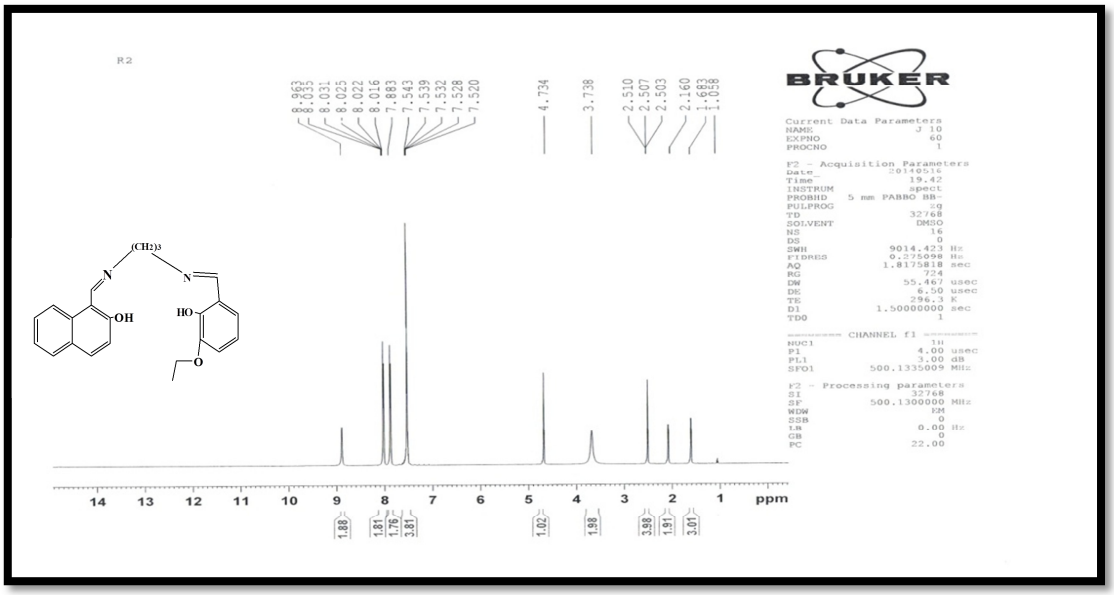
Fig(2) IR spectrum of Schiff base(L_2)



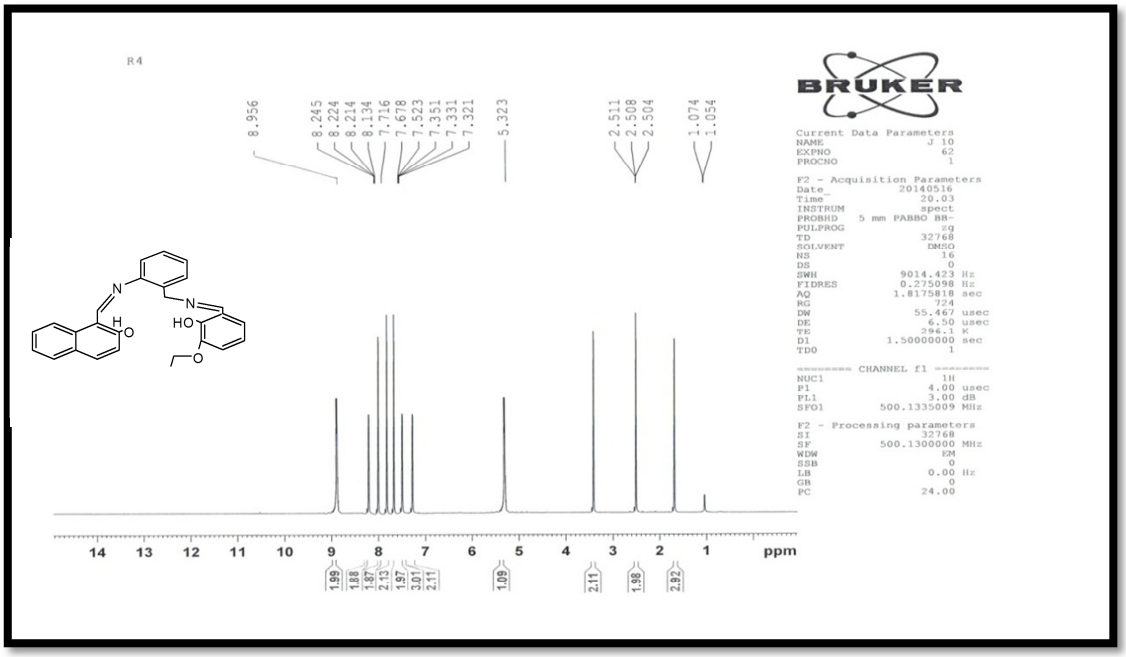
Fig(3) IR spectrum of Schiff base(L_3)



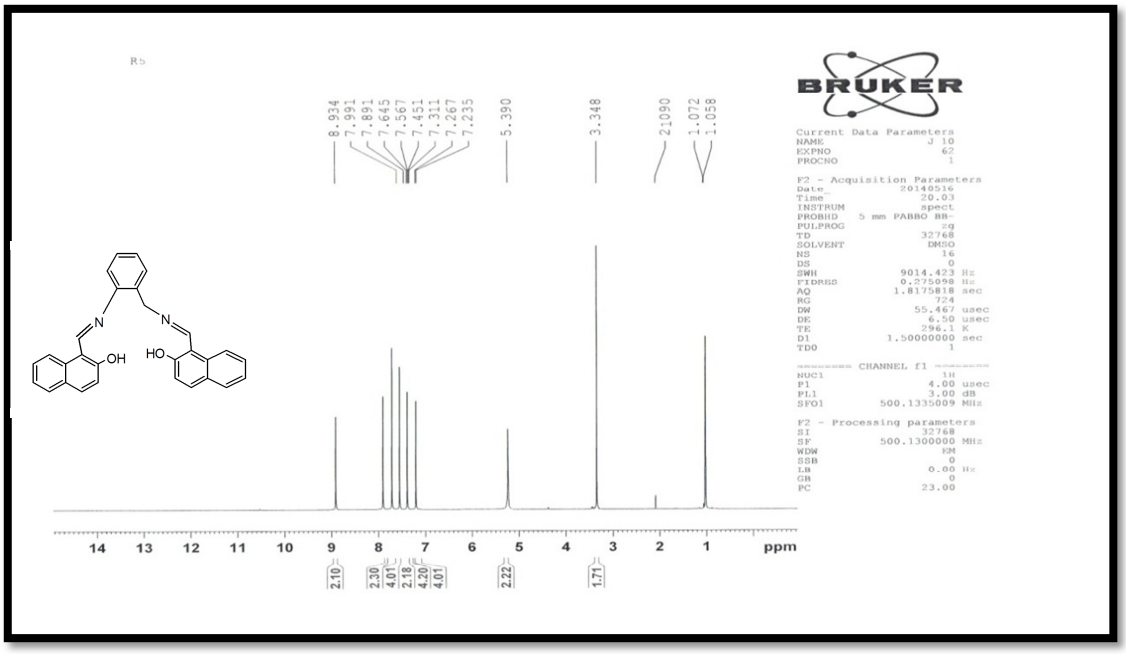
Fig(4) IR spectrum of Schiff base(L_4)



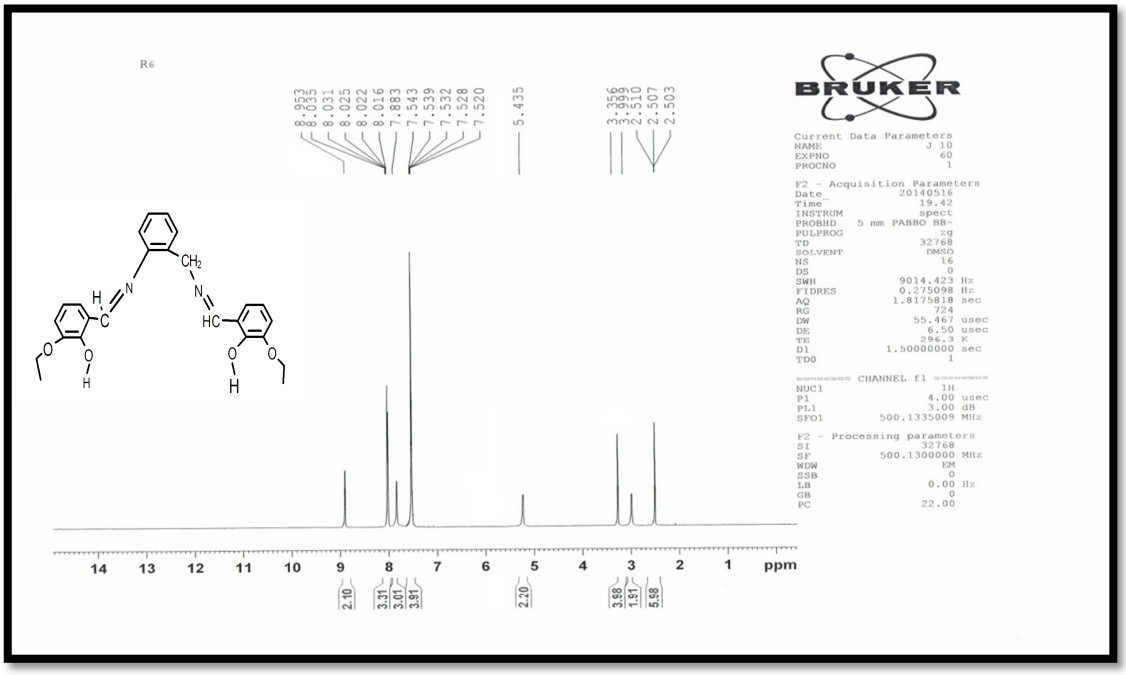
Fig(5) NMR spectrum of Schiff base(L_1)



Fig(6) NMR spectrum of Schiff base(L_2)



Fig(7) NMR spectrum of Schiff base(L_3)



Fig(8) NMR spectrum of Schiff base(L_4)

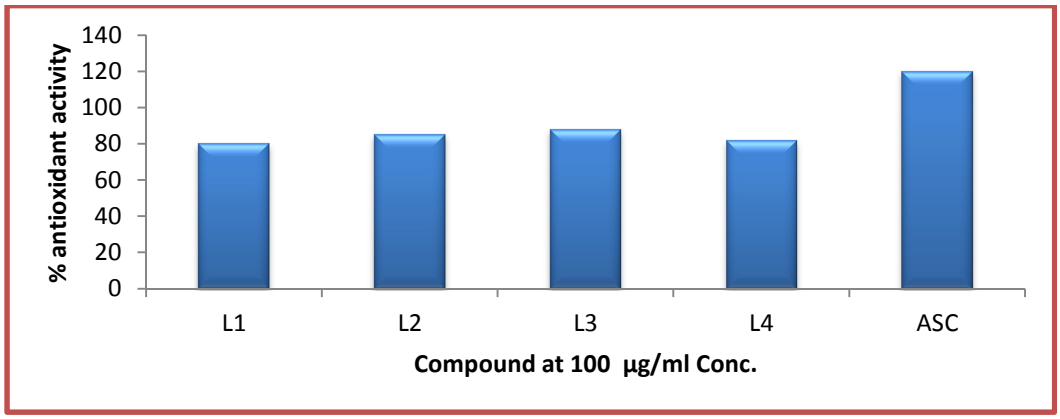


Fig.(9) antioxidant activity of Schiff bases (L_1-L_4) and ASC

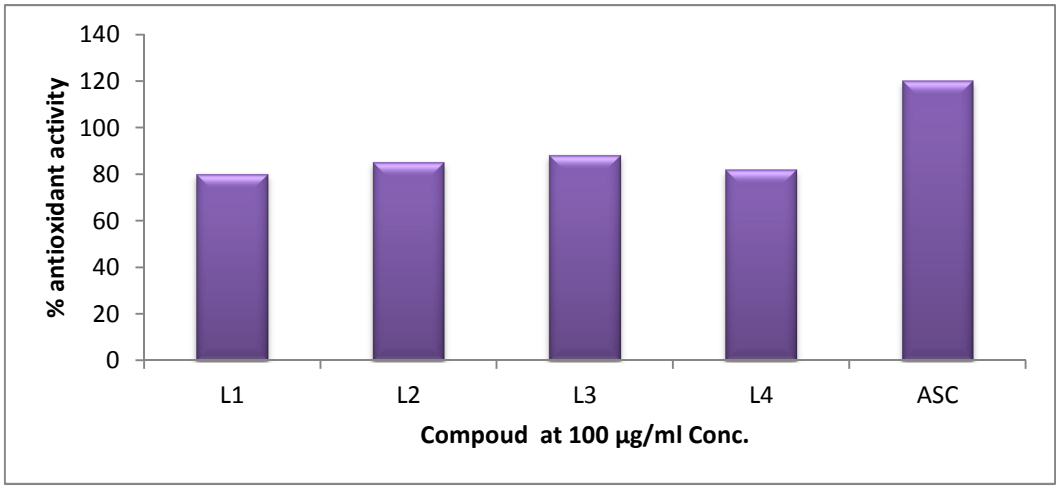


Fig.(10) antioxidant activity of Schiff bases (L_1-L_4) and ASC

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