



## Determination The Structure of New Some Imines by Physical and Chemical Methods



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### Abstract

This paper is concerned with the preparation of eight new imines by standard methods; These having syn oxime and Schiff base structures. They are derived from aromatic aldehydes with suitable primary amines, depending on their structures. This investigation deals with studying structure of imines by chemical and physical methods, (I.R, U.V and <sup>1</sup>H NMR) spectra and melting points. This studying led to diagnose the intra and inter molecular hydrogen bonding in these imines, using dilution method by the aid of I.R spectra. Moreover, the influences of hydrogen bonds on the association process in these imines and their relationship with melting points were also determined

**Key words:** Structure, imines, Schiff bases, I.R., U.V. and <sup>1</sup>H NMR spectra, hydrogen bonding and physical-chemical methods.

### 1. Introduction

Schiff bases are compounds containing an azomethine group (-CH=N-), which formed by the condensation of a primary amine with a carbonyl compound as aldehyde. Aliphatic aldehydes Schiff bases were finding in comparison unstable and were readily polymerizable whereas those of aromatic aldehydes, having an efficient conjugation system, were more stable [1] by resonance. [2]

During the latest few years, a great numbers of aliphatic, aromatic imine as oxime, and their mixture were prepared by Azzouz et al. [3] Azomethines derived from various carbonyls,  $\beta$ -Ketone,  $\beta$ -Ketoester and primary amines were well known compounds had studies extensively by saeed et al. [4]

Several Schiff bases were prepared from salicylaldehyde,[5,6,7] 2-hydroxy-1-naphthaldehyde[8], 2,4-dihydroxybenzaldehyde[9], and benzaldehyde, [10] Schiff bases were known to be neoplasm inhibitors[11], and plant growth regulated[12] Schiff-bases derived from anilines and its derivative with aromatic, aldehydes have a variety of applications in analytical[13] chemistry pharmacological[14] and medical[15]

activities including antimicrobial, anti-inflammatory, anticancer, anti-fungal, anti-tuberculosis, and anti-oxidant.[16] Schiff base complexes[17] had greater importance than the parent Schiff base in biology because the ligands and metal ion can produce the highly active compounds and these interesting application were encourage the worker in this study to increase experimentation in the field of imine

chemistry in the direction of synthesis of a new oxime and Schiff bases and study their structures by physical and chemical methods. Schiff bases and their metal complexes had clear advantages and wonderful applications also they are very important as catalysts in various fields in biological systems, polymers, dyes, in the food, in analytical chemistry, medicinal and pharmaceutical fields.<sup>[18,19]</sup>

### 2. Experimental

#### 2.1. Materials

All chemicals were supplied from Fluka or BDH chemical companies.

#### 2.2. Instrumentation

The U.V spectra of all imines (Schiff bases and oximes) were measured by double beam computerized U.V. (1650 Shimadzu U.V.-Visible

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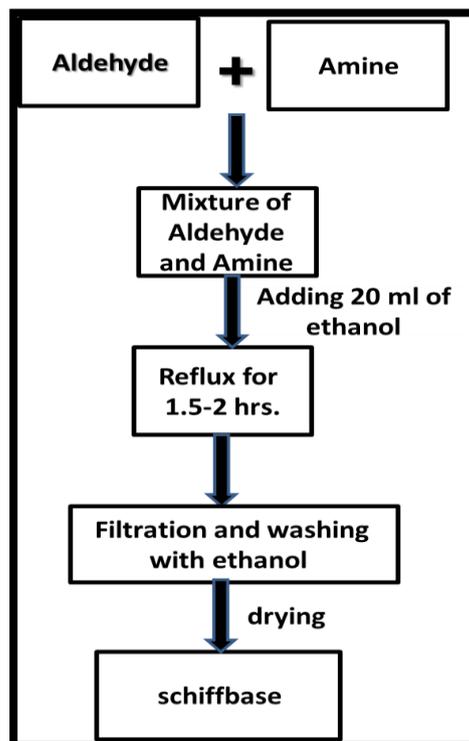
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spectrophotometry) using quartz cuvette of dimensions  $1 \times 1 \times 3 \text{ cm}^3$ . The I.R spectra of liquid and solid of imines had measured by using computerized FTIR Bruker (Tensor 27) spectrophotometer. The melting points of solid imines were measured by using electro thermal melting point (305 m.p. Bibby scientific limited). The  $^1\text{H NMR}$  spectra of all imines were measured by Bruker BioSpin GmbH (400.22 Hz),  $^1\text{H NMR}$  Spectra have been measured on a 400.22 Hz spectrometer using (DMSO) as solvent.

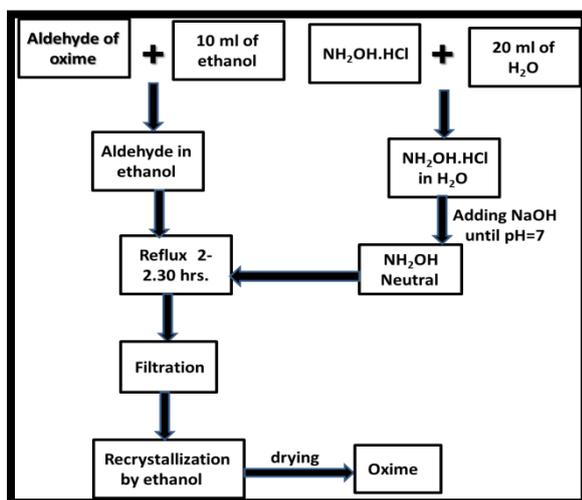
The Sensitive balance to weight the substances type GR-200.

### 2.3. Synthesis of Schiff bases

New imines under investigation were prepared by standard method.<sup>[20]</sup> By mixing  $10^{-2}$  mole amount formaldehyde (p-aminobenzaldehyde, O-aminobenzaldehyde, 3,4-dihydroxybenzaldehyde, cinaaldehyde, with  $10^{-2}$  M Primary amine (hydroxyl) amine, HCl, Aniline, p-chloroaniline, p-nitroaniline, p-toluidine, m-hydroxyaniline) in ethanol solvent, according to standard method. These mixtures were refluxed for 1.5-2 hours. The precipitate was removed from reaction mixture by filtration wash with added ethanol and drying. As shown in the Flow Chart (1) and the Flow Chart (2). Pure imines were purified by re crystallization from absolute ethanol. The number, nomenclature, structure and melting points of pure solid. Samples were shown in table (1).

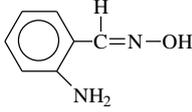
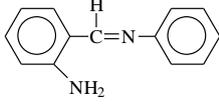
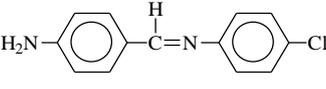
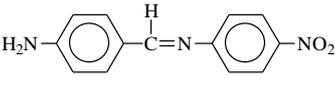
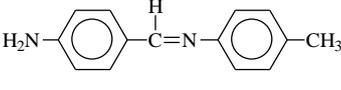
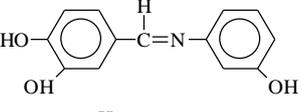
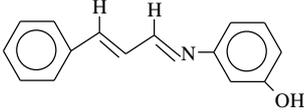


Flow Chart (2): Show preparation process of Schiffbase.



Flow Chart (1): Show preparation process of Oxime.

**Table (1): Shows the number, nomenclature, structures and melting points of new imines**

Com p. No.	Symbol of comp.	Nomenclature	Structure	M.P(°C)
1	Syn-p-ABox	Syn-p-aminobenzaldoxime		180-184
2	Syn-o-ABox	Syn-o-aminobenzaldoxime		162.6
3	o-ABA	o-Aminobenzylideneaniline		170
4	p-AB-p-CA	p-Aminobenzylidene-p-chloroaniline		67-68
5	p-AB-p-NA	p-Aminobenzylidene-p-nitroaniline		144-146
6	p-AB-p-MA	p-Aminobenzylidene-p-methyl aniline		40-42
7	3,4-DHB-m-HA	3,4-dihydroxybenzylidene-m-hydroxyaniline		-
8	C-m-HA	Cinnamildene-m-hydroxyaniline		254-259

### 3. Results and Discussion

#### 3.1. Physical methods

At the beginning of this investigation, it was thought of great importance to confirm the structures of imines under study by physical method. The physical property was often used to identify new compounds or check the purity of samples this encourage the worker in this paper to extend diagnosis of these compounds by the available physical. (U.V., FTIR,  $^1\text{H}$ NMR and melting point)<sup>[18]</sup> and chemical method.

##### 3.1.1. Melting point

It is well known in literature<sup>[21]</sup> that melting points of pure chemical samples were affected by

chemical structure and the ability of hydrogen bond formation especially of intermolecular type. Coming back to table (1) shows a maximum melting point are observed for imines symbol syn p-ABox and CA-m-HA, having melting point values 180-184°C and 254-259°C respectively. These support the presence of intermolecular hydrogen bonding and a high molecular weight of value 210 g/mole in imines(8). On the contrary to that imine syn o-ABox. Had a lower<sup>[21]</sup> melting point of value 162.6 °C due to the presence of intra molecular hydrogen bonding. Meanwhile, imine o-ABA had moderate melting point of value 170°C owing to presence of intra molecular hydrogen bonding. In imine p-AB-p-CA contains amino and chloro groups on aldehyde and amine regions respectively. Chloro group had a great tendency of electron

withdrawing property resulted to an electron deficiency on  $\text{NH}_2$  group. The latter may result to decrease the possibility of hydrogen bond formation, which resulted to a decrease the possibility of polymerization process in, this imine. point of values  $144\text{--}146^\circ\text{C}$ . Schiff base p-AB-p-MA has a low m.p of value  $40\text{--}42^\circ\text{C}$  due to the weak inter molecular hydrogen bonding formation by  $\text{NH}_2$  group, this resulted to low m.p for the Schiff base. Schiff base 3,4-DHB-m-HA has undetermined m.p or exists in liquid state. In other words, this Schiff base exists as a tautomeric mixture of keto and enol forms as in the following tautomerism reaction of type:

Any one of these equilibrium form can act as an impurity with respect to the other form. Therefore, it will not surprise to decrease the melting point of tautomeric enol and keto forms or the existence of

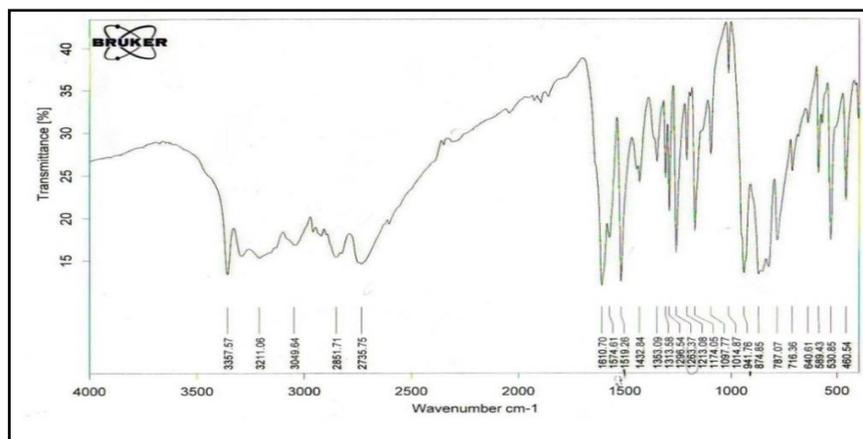
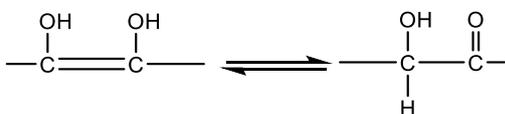


Fig (1): IR spectrum of solid Syn-p-AB-

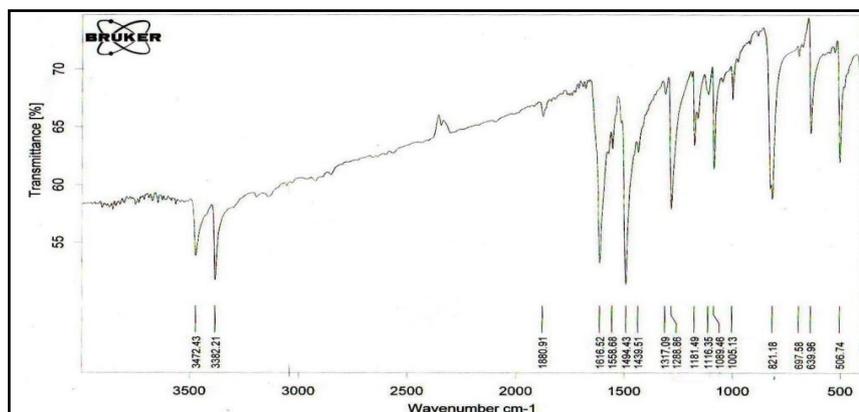


Fig (2): IR spectrum of solid p-AB-p-

Imine p-AB-p-NA contains two opposing electron properties of  $\text{NH}_2$  and  $\text{NO}_2$  groups with intermolecular hydrogen bonding of  $\text{NH}_2$  group, resulted to expected melting

this Schiff base in liquid state. Finally, imine 3,4-DHB-m-HA contains three phenol groups. These resulted in a strong hydrogen bond formation of polymeric<sup>[22]</sup> material of melting point value higher than  $350^\circ\text{C}$ , which was difficult to be measured by normal melting point apparatus

### 3.1.2. The IR Spectra of imines

The I.R spectra of imines (1-8) were measured in solid state and the results were summarized in table (2). Typical I.R spectral for eight solid imines is show in figures (1-5).

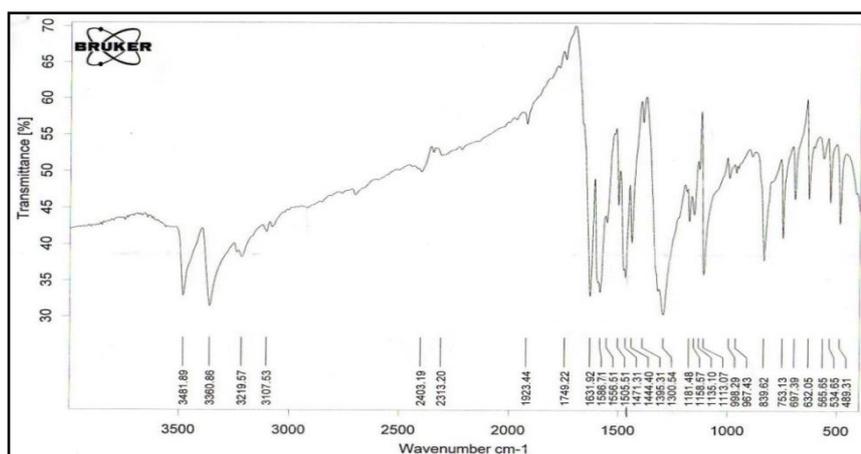


Fig (3): IR spectrum of solid p-AB-p-

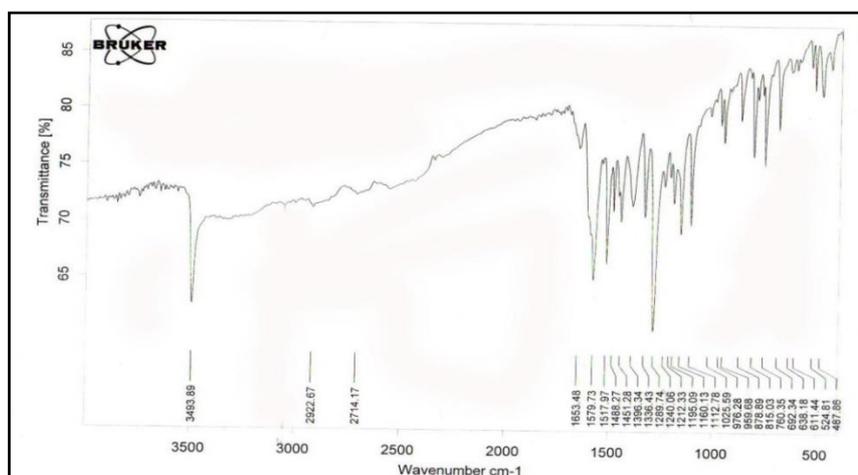


Fig (4): IR spectrum of solid 3,4-DHB-m-

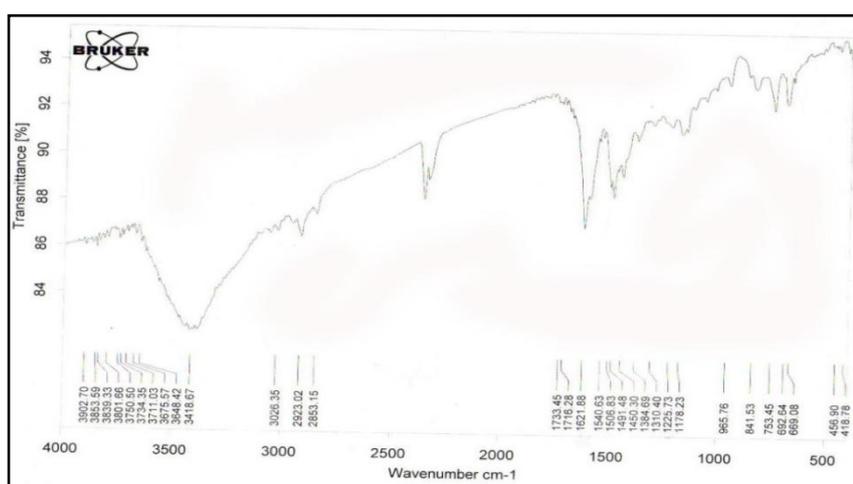


Fig (5): IR spectrum of solid C-m-

These showed the following vibrations: A sharp or a medium stretching wave number in the range (3648.42-3311.26)  $\text{cm}^{-1}$ , which confirm free OH, groups in these imines. The appearance of asymmetric stretching absorption of primary amine group in the range (3481.89-3357.34)  $\text{cm}^{-1}$  of different intensities. Also the appearance of symmetric stretching absorption bands of sharp or medium intensities. Both asymmetric and symmetric bands mentioned confirmed a primary  $\text{NH}_2$  groups were present in imines in the table. A stretching hydrogen bonding bands of medium or broad intensities were present in imines in the table. These were of type H-N...H. In the range (3061.53-2915.38)  $\text{cm}^{-1}$  a weak absorption bands for stretching C-H groups are observed. In the range (1600.92-1494.43)  $\text{cm}^{-1}$  a strong or medium stretching absorptions for aromatic amines. In the range (1668.17-1601.21)  $\text{cm}^{-1}$  a strong stretching absorptions for imine linkage of type  $\text{CH}=\text{N}$  was observed.

A strong stretching absorption for C-Cl band, between (821.18-506.74)  $\text{cm}^{-1}$ . This confirmed the presence of Cl atom in p-AB-p-CA, a very strong wag absorption for  $\text{CH}_2\text{-X}$  band in (1089.43)  $\text{cm}^{-1}$ . In imine (5) a very strong band for asymmetric stretch absorption of  $\text{NO}_2$  group was seen in (1556.51)  $\text{cm}^{-1}$  and a very strong band for symmetric stretch for  $\text{NO}_2$  in (1300.54)  $\text{cm}^{-1}$  was seen. All oximes under study showed tautomerism of oxime to nitroso group ( $\text{N}=\text{O}$ ), with a strong medium intensity band in a range of value (1521.70-1519.26)  $\text{cm}^{-1}$ . These tautomerism studies were in agreement with literature<sup>[5,23,24,25]</sup>. In imine (6), a weak stretching absorption for C-H alkyl in (2915.38)  $\text{cm}^{-1}$  and a symmetric aliphatic C-H bending of methyl group in (1440.82)  $\text{cm}^{-1}$  and symmetric aliphatic C-H bending in (1332.17)  $\text{cm}^{-1}$ . In imine (8), a strong stretching absorption for  $\text{C}=\text{C}$  in (1540.63)  $\text{cm}^{-1}$ , which confirmed to ( $\text{C}=\text{C}$ ) group in conjugation with.

**Table (2): Shown the I.R ( $\text{cm}^{-1}$ ) absorption for imines numbers (1-8) in solid state  $\text{CH}=\text{N}$ .**

$\nu\text{O-H}$	$\nu\text{NH}_2\text{asymetric}$	$\nu\text{NH}_2\text{symmetric}$	$\nu$ hydrogen bond	$\nu\text{C-H}$	$\nu\text{Aromatic}$	$\nu\text{C}=\text{N}$	$\nu\text{Cl}$	$\nu\text{CH}_3$	$\nu\text{NO}_2$
3211.26(s)	3357.34(s)	3290.72(m)	-	3049.64(w)	1574.61(s)	1610.70(s)	-	-	-
3363.50(m)	3460.95(s)	3381.60(s)	3230.79(m)	3046.15(w)	1600.92(v(s))	1668.17(s)	-	-	-
-	3461.10(m)	3313.94(m)	3199.56(m)	3053.84(w)	1581.46(v(s))	1601.21(s)	-	-	-
-	3471.98(s)	3382.21(s)	-	3045.0(w)	1494.43(v(s))	1616.52(v(s))	1089.43(v(s))	-	-
-	3481.89(m)	3360.86(m)	3107.53(b)	3046.0(w)	1586.71(s)	1631.92(s)	-	-	1556.51(s)
-	3450.10(m)	3350.66(s)	3173.42(b)	2915.38(w)	1581.37(s)	1660.07(s)	-	1440.82(s)	-
3493.89(v(s))	-	-	-	3038.46(w)	-	-	-	1332.17(s)	-
3648.42(m)	-	-	3418.67(b)	3061.53(w)	1579.73(v(s))	1653.48(m)	-	-	-
-	-	-	-	3026.35(w)	150683(m)	1621.88(s)	-	-	-

These results, encouraging the worker to repeat the I.R. [23] in the liquid state. The latter was widely used

for study the type of hydrogen bonding in imines (2,3) which have  $\text{NH}_2$  group in ortho position. Typical I.R spectra for imines (2,3) shown in figures (6-8).

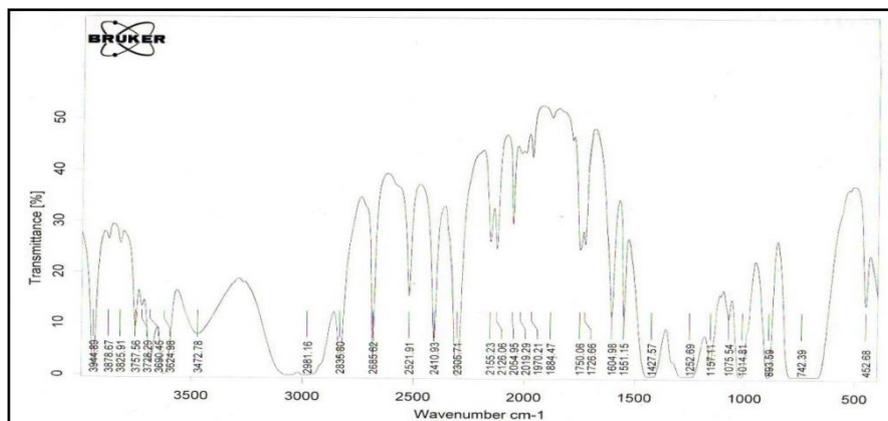


Fig (6): The IR spectra of  $10^{-3}$  M of imine (2) before the dilution in

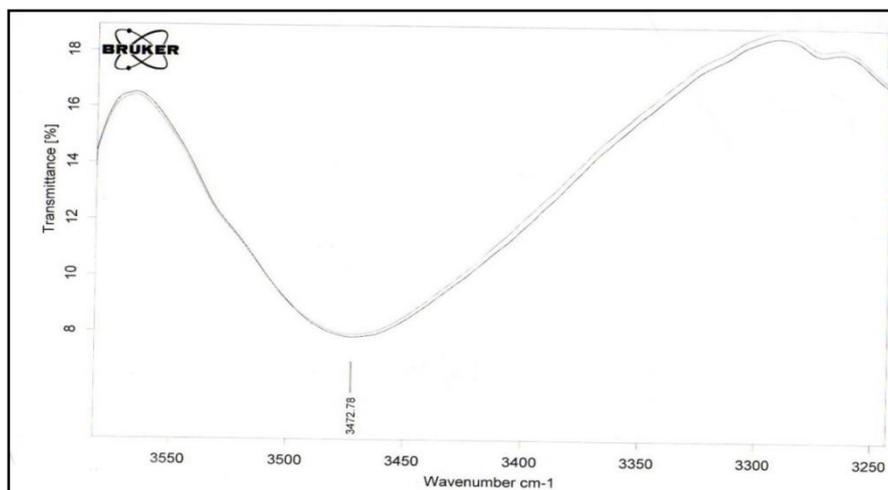


Fig (7): The IR spectra of  $10^{-3}$  M of imine (2) after the dilution in Dichloromethane

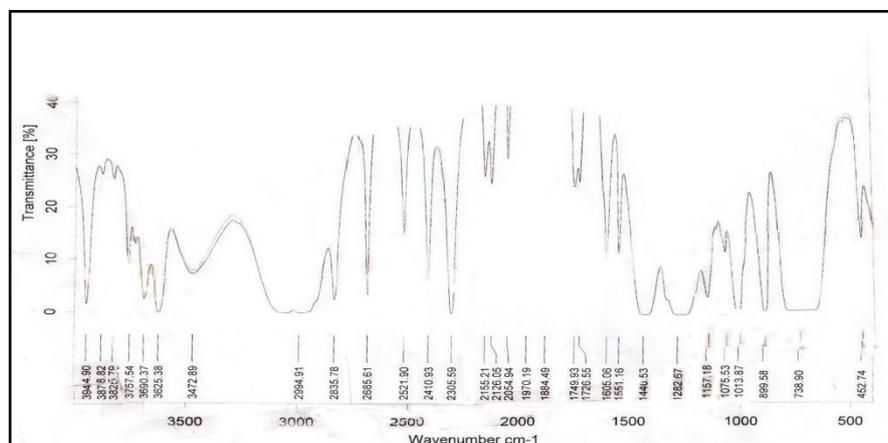


Fig (8): Show the IR spectra of  $10^{-3}$  M imine (3) before and after the dilution

### Some I.R spectra of imines in liquid state

Actually all imines under study had either  $\text{NH}_2$  or OH group. These groups had an ability to form either intermolecular or intra molecular hydrogen bonding.

Such type of bonding was examined by dilution of  $10^{-3}\text{M}$  solutions of ratio 1:3 in 1,2-dichloromethane. Results collected were shown in figures (6-8).

Figures (6-8) were confirmed the existence of intra molecular and intermolecular hydrogen bonding in imines o-A Box and o-ABA respectively. imines 1 and 2 were studied in which the type of hydrogen bonding was inter or intra due to their containment of the amino group in ortho site, using an non polar solvent, so the dilution process is able to distinguish whether the hydrogen bond is inter or intra, so the intra hydrogen bond is

affected by intensity to a less degree than the inter hydrogen bond. Other imines had intermolecular hydrogen bonding the result agreement with other similar studies in this field<sup>(24,26,27)</sup>.

### 3.1.3. U.V spectra:

The U.V spectra of imines (1-8) show bands in table (3) with molar extension coefficient values of more than 1000 in unit  $\text{L.mole}^{-1}.\text{cm}^{-1}$ <sup>(21,28)</sup>. These were interpreted by  $\pi \rightarrow \pi^*$  transition<sup>(21)</sup> which can identify for knowing molar extension coefficient by using beer-Lambert law. All imines have  $\epsilon_{\text{max}}$  value greater than one thousand. These were measured in polar solvent (ethanol), these measurements were in agreement with the previous imines (1-8). Were measured electronic spectra in polar solvent (ethanol).

Table (3): Show U.V spectra of imines in (1-8) ethanol

No.	Symbol of imine	$\lambda_1$	$\epsilon_{1\text{max}}$	$\lambda_2$	$\epsilon_{2\text{max}}$	$\lambda_3$	$\epsilon_{3\text{max}}$
		nm	$\text{L.mole}^{-1}.\text{cm}^{-1}$	nm	$\text{L.mole}^{-1}.\text{cm}^{-1}$	nm	$\text{L.mole}^{-1}.\text{cm}^{-1}$
1	Syn-p-ABox	282	18920	219	13050	-	-
2	Syn-o-ABox	293	5940	224.2	4775	-	-
3	o-ABA	291	53320	220.8	26320	-	-
4	p-AB-p-Ca	300.8	2882	243.4	16310	-	-
5	p-AB-p-Na	372	10263.1	355.5	8652.63	367.5	9168.42
6	p-AB-p-MA	326.6	45.500	236.0	29250	-	-
7	3,4-DHB-M-Ha	314	1224	280.6	1368	233.2	1850
8	C-M-Ha	268.5	14185	266.5	14260	-	-

A  $\pi \rightarrow \pi^*$  transition have identified for all imines from  $\epsilon_{\text{max}}$  value of greater than one thousand in unit  $\text{L.mole}^{-1}.\text{cm}^{-1}$ .

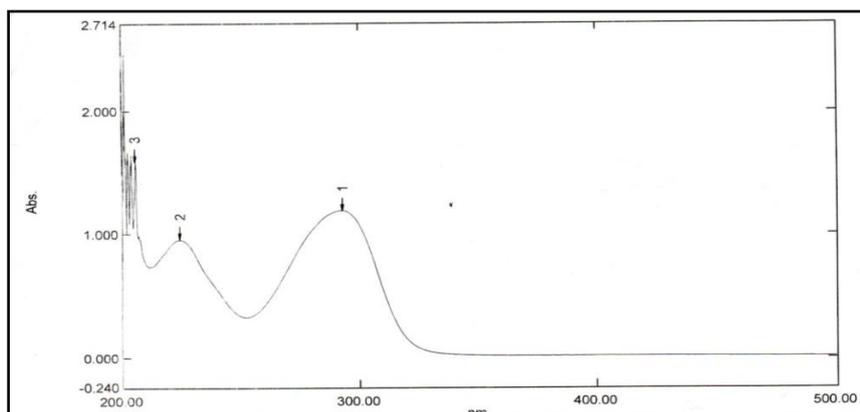
Table (3) and Figures (9-14) of U.V spectra for imines (1-8) showed the following:

bonding in imines o-A Box and o-ABA respectively. imines 1 and 2 were studied in which the type of hydrogen bonding was inter or intra due to their containment of the amino group in ortho site, using an non polar solvent, so the dilution process is able to distinguish whether the hydrogen bond is inter or intra, so the intra hydrogen bond is affected by intensity to a less degree than the inter hydrogen bond. Other imines had intermolecular

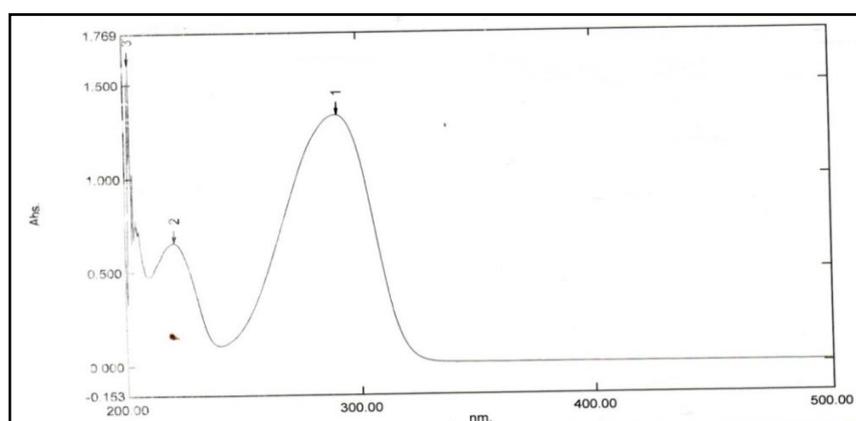
hydrogen bonding the result agreement with other similar studies in this field.<sup>[24,26,27]</sup>

Imines (1, 2, 3, 4, 6, 8) showed two U.V. bands. These mean the existence of imines in two different planes. The first one is assigned to aromatic ring originated from aldehyde part of molecule.

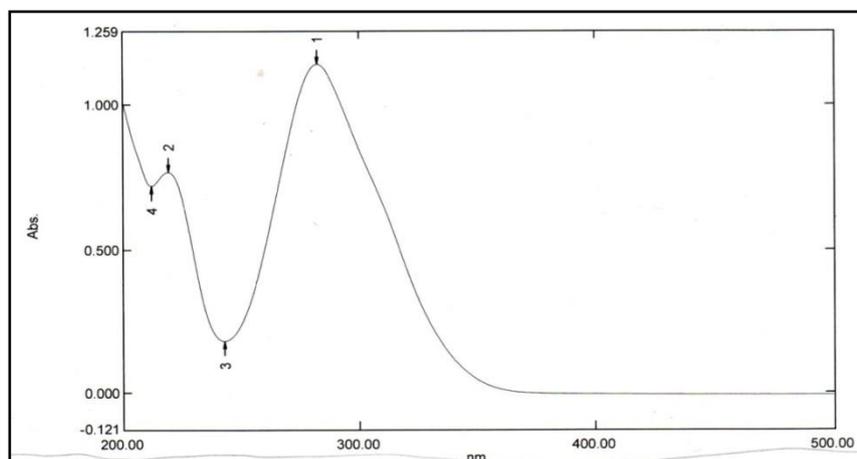
The second one belonged to rest molecule. Imines (5,7) on the other hand showed three bands. These were assigned to aldehyde ring, azomethine linkage and primary amine ring respectively.



**Fig (10):** Show the U.V spectra of imine (2) in Ethanol



**Fig (11):** Show the U.V spectra of imine (3) in Ethanol



**Fig (9):** Show the U.V spectra of imine (1) in Ethanol

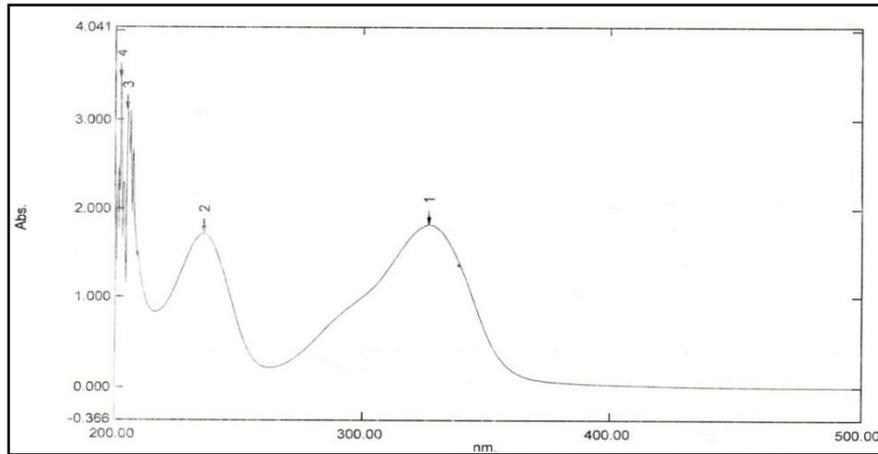


Fig (13): Show the U.V spectra of imine (6) in Ethanol

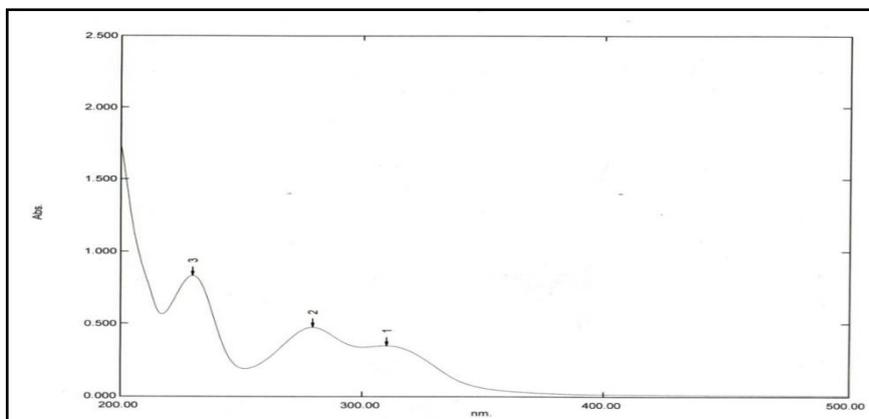


Fig (14): Show the U.V spectra of imine (8) in Ethanol

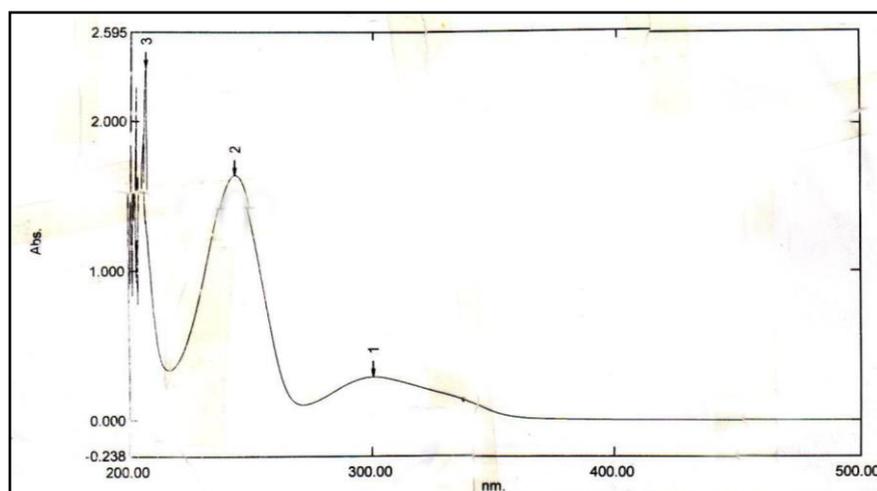


Fig (12): Show the U.V spectra of imine (4) in Ethanol

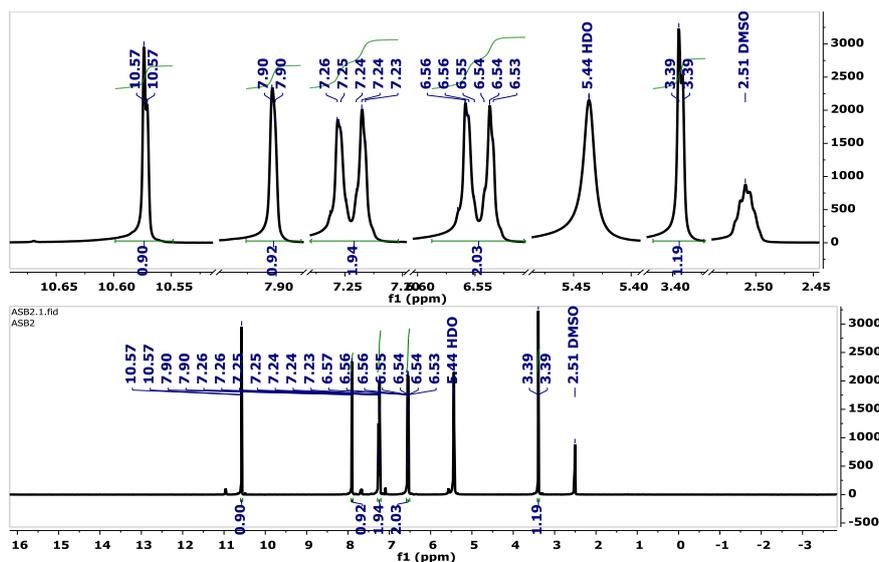


Fig (15): Show the  $^1\text{H}$  N.M.R spectra of imine (1)

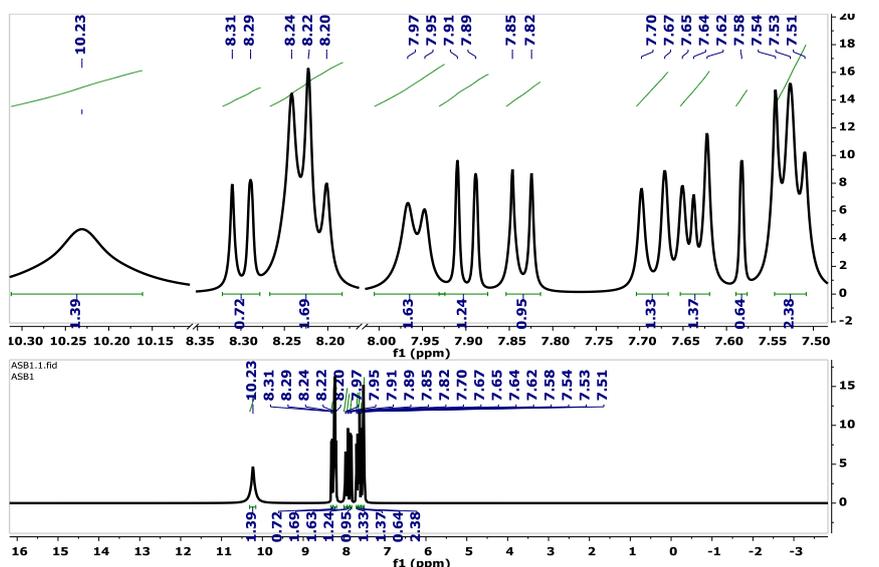


Fig (16): Show the  $^1\text{H}$  N.M.R spectra of imine (8)

### 3.1.4. $^1\text{H}$ NMR spectra:

The  $^1\text{H}$ NMR spectra of imines (1-8) were measured and showed the following:

Imine(1): (s,1H,OH) 10.57, (s,1H,C=N) 7.9,(d,4H,Ar.) 7.26-6.53, (s,2H,NH<sub>2</sub>) 3.40 as in fig.(15).

Imine (2): (s,1H,OH) 12.09, (s,1H,C=N) 7.9, (m,4H,Ar.) 7.63-6.54, (s,b,2H,NH<sub>2</sub>) 5.93.

Imine(3): (s),(d,4H,Ar.) 7.64-7.61, (m,5H,Ar.) 6.57- 6.53, (s,b,2H,NH<sub>2</sub>) 5.89.

Imine (4): (s,1H,C=N) 8.32, (d,4H,Ar) 7.61-7.39, (d,4H,Ar.) 7.20-6.62) (s,2H,NH<sub>2</sub>) 5.91.

,Ar.) 7.91-7.51 as in fig.(16).

Imine(5): (s,1H,C=N) 10.32, (d,4H,Ar.) 8.85-8.26 (d,4H,Ar.) 7.69-8.02, (s,2H,NH<sub>2</sub>) 5.91.Imine (6): (s,1H,C=N)8.66, (d,4H,Ar.) 7.23-7.21, (d,4H,Ar.) 7.25-6.55, (s,2H,NH<sub>2</sub>) 3.32 (s,3H,CH<sub>3</sub>) 2.24,.

Imine(7): (s,1H,C=N) 8.45, (s,2H,OH) 14.79 (s,1H,OH) 10.13, (s,2H,Ar.) 6.4-7.62 (d,4H,Ar.) 12.01-12.4, (t,1H,Ar) 7.39

. Imine (8): (d,1H,C=N) 8.31-8.29, (s,1H,OH) 10.23, (t,H,C=C) 8.24-8.20, (d,H,C=C) 7.97-7.95, (m,4H

### 3.2. Chemical methods <sup>[27]</sup>:

These were accomplished by using standard procedures for testing functional groups in imines listed in table (1). These showed tests for the following groups: (Primary aromatic amine, Oxime group, Aromatic nitro group, Phenol group and Aliphatic alkene C=C).

All these tests displayed in table(4) when applied on imines (1-8) listed in table (1), showed a positive result, which support the correct synthesis of imines under study.

All these results were in full agreement with other similar studies in this field. <sup>[29,30]</sup>

**Table (4): Show the chemical test of imine (1-8).**

No. of imine	Ferric chloride test	Diazotization test	Bromine water test	Ferrous hydroxide test
1	(Violent color) +ve	+ ve	-	-
2	(Violent color) +ve	+ ve	-	-
3	-	+ ve	-	-
4	-	+ ve	-	-
5	-	+ ve	-	+ve (red brown) precipitate
6	-	+ ve	-	-
7	(blue) +ve	-	-	-
8	(blue) +ve	-	+ve decolorization of bromine solution	-

### 4. Conclusions

All new imines were prepared by standard method of reaction of aldehyde with primary amine and hydroxyl amine, the structure of all imines was confirmed by chemical method (test) with positive result and physical method, the physical methods contained melting points and spectroscopic methods ,which it included I.R, U.V and H<sup>1</sup>NMR spectra, the I.R spectra was used in the liquid state to distinguish the intra hydrogen bond from inter, the presence of intra hydrogen bond of some imine worked to raise the melting point of these compounds, the I.R spectra of imines (1,2) show tautomerism reaction of oxime to nitroso group.

### Conflicts of interest

There are no conflicts to declare

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