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# Synthesis, Characterization and Study of the Biological activity of Some Aldimines Derivatives

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**Abstract:** A number of imines derivatives have been synthesized, they were obtained by condensation of aromatic aldehyde derivatives with primary aromatic amine derivatives. Their structures have been characterised by IR ,  $^1\text{H NMR}$  in addition to the elemental analysis. The biological activity of these imines ( which are also known as Schiff bases ) were examined against different type of microorganisms and they found to have considerable activity in comparison with the most commonly used antibiotics .

**Keywords:** Synthesis, Biological activity, Characterization, Aldimines Derivatives

## Introduction

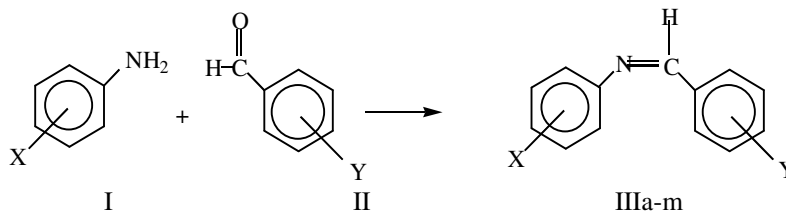
The chemistry of heteropolar unsaturated functions are very well explored <sup>1, 2</sup>, and imines in particular are of special interest in literature due to their numerous practical applications. <sup>3-5</sup> Aldimines have been generally used as substrates in the formation of a large number of industrial compounds via cycloaddition , ring closure , replacement reaction , etc. <sup>6,7</sup>. In addition, the aldimines of heterocyclic carbaldehyde , which are widely used in the production of pharmaceuticals have taken an important place among the compounds of biological interest and the benzylidene derivatives display a large variety of activities. <sup>8,9</sup> Imines and their complexes have a variety of applications in biological, clinical and analytical fields. <sup>10-12</sup>

## Experimental

All chemicals and solvent were obtained from Aldrich,  $^1\text{H}$  NMR spectra were recorded with a Bruker AC 300 spectrophotometer using  $\text{CDCl}_3$  and reported relative to TMS as internal standard. IR spectra were recorded on FT - IR Spectrometer, Perkin- Elmer using KBr pellets.

### General procedure for preparation of Imines

The imines (or Schiff bases) were prepared by a modification of the reported methods.<sup>13 - 16</sup>



III <sub>a</sub>	X= H	X=Y
III <sub>b</sub>	X= H	Y= Br ( <i>p</i> )
III <sub>c</sub>	X= N(CH <sub>3</sub> ) <sub>2</sub> ( <i>p</i> )	Y= H
III <sub>d</sub>	X= OH ( <i>m</i> )	Y= NO <sub>2</sub> ( <i>p</i> )
III <sub>e</sub>	X= OH ( <i>m</i> )	Y= H
III <sub>f</sub>	X= OCH <sub>3</sub> ( <i>p</i> )	Y= OCH <sub>3</sub> ( <i>p</i> )
III <sub>g</sub>	X= OCH <sub>3</sub> ( <i>p</i> )	Y= CH <sub>3</sub> ( <i>m</i> )
III <sub>h</sub>	X= OH ( <i>m</i> )	Y= OH ( <i>p</i> )
III <sub>i</sub>	X= OCH <sub>3</sub> ( <i>p</i> )	Y= H
III <sub>j</sub>	X= OCH <sub>3</sub> ( <i>o</i> )	Y=NO <sub>2</sub> ( <i>p</i> )

### A typical procedure for the synthesis of imines is as follows

A solution of 0.01mole of the amine ( dissolved in 50 ml ethanol ) was slowly added to a solution of 0.01 mole of the aldehyde ( in 50 ml ethanol ).After stirring the reaction mixture for 45 minutes at 40 -50°C, the precipitate was cooled and collected by filtration , then washed several times with distilled water. The products were recrystallised from water – ethanol mixture (1: 1) and air dried .The purity of the products were confirmed by elemental analysis. Details of their physical properties are given in Table 1.

## Results and Discussion

### Spectroscopy Study

#### IR Spectra

In general the Schiff bases exhibits the expected features of the standard IR spectra for this type of compounds. The spectra shows no absorption at about  $1700\text{ cm}^{-1}$  which indicates the absence of free carbonyl group .The strong absorption in the  $1620\text{ cm}^{-1}$  region is assigned to the azomethine group present in the Schiff bases The IR spectral data are given on Table 2 .

**Table 1** Physical Properties of Schiff bases and Elemental Analysis

Compound No.	Molecular Formula	M.P °C	yield %	C %	H %	N %
III <sub>a</sub>	C <sub>13</sub> H <sub>11</sub> N	51	85	86.18 (86.89)	6.07 (5.43)	7.73 (8.19)
III <sub>b</sub>	C <sub>13</sub> H <sub>10</sub> NBr	78	74	60.00 (59.16)	3.85 (3.87)	5.38 (6.50)
III <sub>c</sub>	C <sub>15</sub> H <sub>16</sub> N <sub>2</sub>	67	83	80.35 (79.16)	7.14 (6.65)	12.50 (11.95)
III <sub>d</sub>	C <sub>13</sub> H <sub>10</sub> N <sub>2</sub> O <sub>3</sub>	75	91	64.46 (63.26)	4.13 (4.23)	11.57 (10.71)
III <sub>e</sub>	C <sub>13</sub> H <sub>11</sub> NO	72	98	79.19 (78.51)	5.58 (5.32)	7.11 (6.51)
III <sub>f</sub>	C <sub>15</sub> H <sub>15</sub> NO <sub>2</sub>	145	94	74.69 (75.64)	6.22 (6.09)	5.81 (5.93)
III <sub>g</sub>	C <sub>15</sub> H <sub>15</sub> NO	54	98	80.00 (80.90)	6.67 (6.94)	6.22 (6.11)
III <sub>h</sub>	C <sub>13</sub> H <sub>11</sub> NO <sub>2</sub>	175	97	73.23 (72.28)	5.16 (5.12)	6.57 (5.95)
III <sub>i</sub>	C <sub>14</sub> H <sub>13</sub> NO	44	97	79.62 (79.56)	6.16 (6.12)	6.63 (6.81)
III <sub>j</sub>	C <sub>14</sub> H <sub>12</sub> N <sub>2</sub> O <sub>3</sub>	88	87	65.62 (65.25)	4.68 (4.28)	10.93 (10.8)

**Table 2** IR Spectra of the Schiff bases ( in cm<sup>-1</sup>)

Compound No.	v(O-H)	v(CH <sub>3</sub> ), v(OCH <sub>3</sub> )	v(NO <sub>2</sub> )	v(C=N)	v(C-Br)	v(C-O)	v(C-N)	v(C-H) arom	v(C-H) alkene
III <sub>a</sub>	-	-	-	1624	-	-	1300 1365	691 760	3059
III <sub>b</sub>	-	-	-	1590	741	-	1281 1312	698 671	3059
III <sub>c</sub>	-	1358 1443	-	1601	-	-	1315 1358	814 760	3030
III <sub>d</sub>	3490 phenol	-	1304 1474	1593 1642	-	1177 phenol	1107 phenol	837 752	3090
III <sub>e</sub>	3414 phenol	-	-	1590	-	1389 phenol	1273 phenol	779 687	3055
III <sub>f</sub>	-	1366 1462	-	1613	-	1169 1246	1296	837	2951 3000
III <sub>g</sub>	-	1454 1373	-	1605	-	1157 ether	1246	826 783	2920 3000
III <sub>h</sub>	3285 3430	-	-	1590	-	1150 1265	1339	833 799	2950 3050
III <sub>i</sub>	-	1450	-	1610	-	1020 1190	1250	840 692	3000
III <sub>j</sub>	-	1460	1515 1320	1600	-	1050 1100	1190	840 755	-

<sup>1</sup>H - NMR

Again the <sup>1</sup>H - NMR spectra shows the expected signals which corresponds to the various groups present on each compounds . The common signals which appears at δ 8.6 and 6.4 - 7.3 ppm , in all compounds were assigned to methine and aromatic protons respectively .The <sup>1</sup>H - NMR spectral data are shown on Table 3.

**Table 3** <sup>1</sup>H-NMR Data of Schiff bases ( δ in ppm)

Compound No.	(H)C=N	Ar-H	OH	OCH <sub>3</sub>	CH <sub>3</sub> -Ar	(CH <sub>3</sub> ) <sub>2</sub> N	(CH=CH)
III <sub>a</sub>	8.61	7.24- 7.94	-	-	-	-	-
III <sub>b</sub>	8.53	7.07- 7.89	-	-	-	-	-
III <sub>c</sub>	8.39	7.16- 7.75	-	-	-	3.00	-
III <sub>d</sub>	7.95	6.58- 7.40	9.90	-	-	-	-
III <sub>e</sub>	8.51	7.21- 7.43	9.69	-	-	-	-
III <sub>f</sub>	8.53	6.94- 7.67	-	3.76- 3.82	-	-	-
III <sub>g</sub>	8.50	6.99- 7.82	-	3.83- 3.86	2.33	-	-
III <sub>h</sub>	8.51	7.17- 7.36	9.90	-	-	-	-
III <sub>i</sub>	8.40	6.89- 7.98	-	3.85	-	-	-
III <sub>j</sub>	8.30	6.88- 7.81	-	3.96	-	-	-

*Biological assays*<sup>17,18</sup>

Anti bacterial activity of the Schiff bases have been carried out against several types of bacteria such as *S.aureus* ; *E.coli.*; *B. Subtilis* and *Staphylococcus* , using nutrient agar medium by well diffusion method .The concentration of samples ranged from 2-4 mg /ml in pure methanol .The cultures were carried out at the Botany department ,College of Science, Garyounis university .The results are expressed in MIC (minimal inhibitory concentration ) solvent blanks were run against each test organism in all assays , the results of the biological activities are shown on Table 4 .

**Table 4** Biological Activity Data

Compound No.	<i>E.coli</i> (mm/Zone)			<i>S.aureus</i> (mm/Zone)		
	0.1	0.01	0.001	0.1	0.01	0.001
III <sub>a</sub>	-	-	-	11	-	-
III <sub>d</sub>	14	8	7	10	6	4
III <sub>e</sub>	12	-	-	-	-	-

## References

- 1 Comprehensive Organic Chemistry , Synthesis and reactions of Organic Compounds , Ed. Sir Derek Barton , Vol. 2 part 8 p.383 , London .
- 2 Patai S , The Chemistry of the ( C = N ) bond , S . Patai , Ed ., Interscience Publisher New York , London, 1970 , Chapter 1 .
- 3 Hadjondis E , *Mol . Eng .* 1995 , **5** , 301 .
- 4 Koll A , *Int . J . Mol . Sci.* 2003 , **4** , 434 .
- 5 Echevarria A, Nascimento M G, Geronimo V, Miller I and Giesbrecht A, *J. Braz . Chem . Soc.*, 1999 , **10** , 60.
- 6 Brown A D and Colvin E V, *Tetrahedron Lett .* 1991, **32** , 5,87.
- 7 Burtwood D A, Gallucci J and Hart D J, *J . Org . Chem .* , 1985 , **50** , 5120 .
- 8 Brown F C , *Chem . Rev .* 1961 , **61** , 463 .
- 9 Raasch M S, *J Heterocyclic Chem .* 1974 , **11** , 587 .
- 10 Singh P, Goel R L, Singh B P, *J . Indian Chem .* , 1975 , **52** , 958 .
- 11 Mahindra A M, Fisher J M and Rabinovitz , *Nature*, 1983 , **303** , 64 .
- 12 Patel P R, Thaker B T , Zele S, *Indian J . Chem .* 1999 , **38A** , 563 .
- 13 Marzilli L G, Marzilli P A and Halpern J, *J . Am . Chem . Soc .* 1971, **93**, 1374.
- 14 West O, *J . Chem . Soc .* 1954 , 395 .
- 15 Bigelow L A and Gatough H , *Org. Synth. Coll.*, 1967, **I** 80 .
- 16 Textbook of Practical Organic Chemistry, Vogel A I, Longmann 1973, London.
- 17 Perez C, Pauli M and Bazevgue P, *Acta Biol . Med . Exp.*, 1990 , **15** , 113 .
- 18 Berry B F, Beezer A E, Miles R J, Smith B V, Miller J, Nascimento M G, *Microbios*, 1986 , **45**, 181.