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# Synthesis ,Characterization and biological activity study of new Schiff's bases containing 3,4-dimethyl maleimide moiety

**Ahlam MaroufAl-Azzawi**

University of Baghdad - College of Science.

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3,4-dimethyl maleimide moiety.

**ABSTRACT**

A series of new Schiff's bases containing 3,4-dimethyl maleimide moiety have been synthesized via multisteps synthesis including reaction of 3,4-dimethyl maleic anhydride with aniline producing N-phenyl-3,4-dimethyl maleimide which react with chlorosulfonicacid producing 4-(N-3,4- dimethyl maleimidyl ) phenyl sulfonyl chloride which on amination with hydrazine hydrate yielded in turn 4- (N-3,4-dimethyl maleimidyl) phenyl sulfonyl hydrazine and this when condensed with various aromatic aldehydes and ketones afforded the desirable Schiff bases.

Structures of the prepared compounds were confirmed by spectroscopic methods including FTIR, 1HNMR,C13NMR spectroscopy and C.H.N analysis. The synthesized Schiff's bases were screened for their antibacterial activity against three microorganisms: Staphylococcus aureus, Escherichia Coli, and Psedomonas aeruginosa. They were found to exhibit high antibacterial activity.

## Introduction

Since the isolation of phyllanthimide a new alkaloid present in phyllanthus sellowianus which possessed antispasmodic activity several synthetic analogues (cyclic imides) have been reported as antibacterial, antifungal, antispasmodic and analgesic (1-4). Cyclic imides such as maleimides(5), phthalimides (6), succinimides, gluterimides and their derivatives(7) contain an imide ring and a general structure

[ -CO-N(R)-CO-] that confers hydrophobicity and neutral characteristic.

A diversity of biological activities and pharmaceutical uses have been attributed to them such as antinociceptive , antionvulsant and antitumor (8,9) .

Moreover a number of researches have reported the using of substituted 3,4- dimethyl maleimides in chemotherapy of tumors, ermatomycosis and candidiasis (10).

On the other hand biocidal activities of Schiff's bases have also been well established (11) thus a variety of Schiff's bases are reported to show a diversity of interesting biological activities including antibacterial, antifungal, antimouse hepatitis virus (MHV), anticancer and herbicidal activities (12,13).

* Corresponding author at: University of Baghdad - College of Science, Iraq.
E-mail address:

Biological activities of these compounds in general have been attributed to the toxophoric (C=N) linkage in their structures(14). Keeping these above facts in view we considered it of interest to synthesize a series of new Schiff's bases containing 3,4-dimethyl maleimide moiety in their structures.

The new compounds were expected to possess biological activity since their molecules were built from two biologically active components.

## Experimental

All chemicals were from BDH,Aldrich and were used without further purification .

Melting points were determined in open capillaries on Thomas Hoover apparatus and were uncorrected. FTIR spectra were recorded on SHIMADZU FTIR- 8400 Fourier Transform Infrared spectrophotometer. 1HNMR and C13NMR spectra were recorded on Bruker spectrospin ultra shield magnets 300MHz instrument in Ahl-Albate University in Jordon using tetramethyl silane (TMS) as an internal standard and DMSO-d6 as a solvent. Elemental analysis (C.H.N) was performed on Perkin-Elmer 240 element analyzer in Jordon .

### 1.Preparation of N-phenyl-3,4-dimethyl maleimide [1]

Literature procedures (15)were used in

preparation of the titled compound with minor modifications.

To a solution of (0.01 mole) of 3,4-dimethyl maleic anhydride in (25mL) of ether ,(0.01 mole) of aniline was added drop wise with stirring and cooling. After standing overnight at room temperature the solvent was evaporated to dryness and the residue was dissolved in (10 mL) of acetone followed by filtration. The imide was precipitated with cyclohexane then purified by recrystallization from cyclohexane Yield 77 % m.p.=90-91 0C.

**2. Preparation of 4-(N-3,4- dimethyl maleimidyl) phenyl sulfonyl chloride [2]**

The titled compound was prepared according to literatures (16) with some modifications.

suitable dry round bottomed flask fitted with dropping funnel (0.01 mole) of In a N- phenyl -3,4-dimethyl maleimide was placed and the dropping funnel was charged with (4 mL) of chlorosulfonicacid. Chlorosulfonicacid was added drop wise during two hours with continuous stirring and keeping temperature at zero 0C .When addition was completed the mixture was stirred at room temperature for (10 hrs) then was poured on crushed ice carefully with stirring.The resulted precipitate was filtered , washed with cold water and dried then purified by recrystallization from methanol Yield 81 % m.p.=165-166 0C .

**3. Preparation of 4-(N-3,4-dimethyl maleimidyl) phenyl sulfonyl hydrazine [3]**

To a solution of (0.004 mole)of compound [2] in (5 mL) absolute ethanol, (0.004 mole) of hydrazine hydrate was added drop wise with stirring and keeping temperature at (-10 ) 0C.

The resulted mixture was refluxed for 3hrs then cooled to room temperature before pouring on crushed ice with stirring .The resulting precipitate was filtered , washed with cold water and dried Yield 75 % m.p.=180-182 0C .

Physical properties and FTIR spectral data of compounds [1],[2] and [3] are listed in Table (1).

**4.Preparation of Schiff's bases [4-15]**

A mixture of 4-(N-3,4-dimethyl maleimidyl) phenyl sulfonyl hydrazine (0.01 mole), aromatic aldehyde or ketone (0.01 mole) and (2-3) drops of glacial acetic acid in absolute ethanol (30 mL)was refluxed for 6 hrs (17).

The solvent was removed under reduced pressure and the residue was poured into cold water. The obtained precipitate was filtered, dried and recrystallized from suitable solvent. Physical properties and FTIR spectral data of the prepared compounds [4-15] are listed in Table (2).

**5. Antibacterial activity**

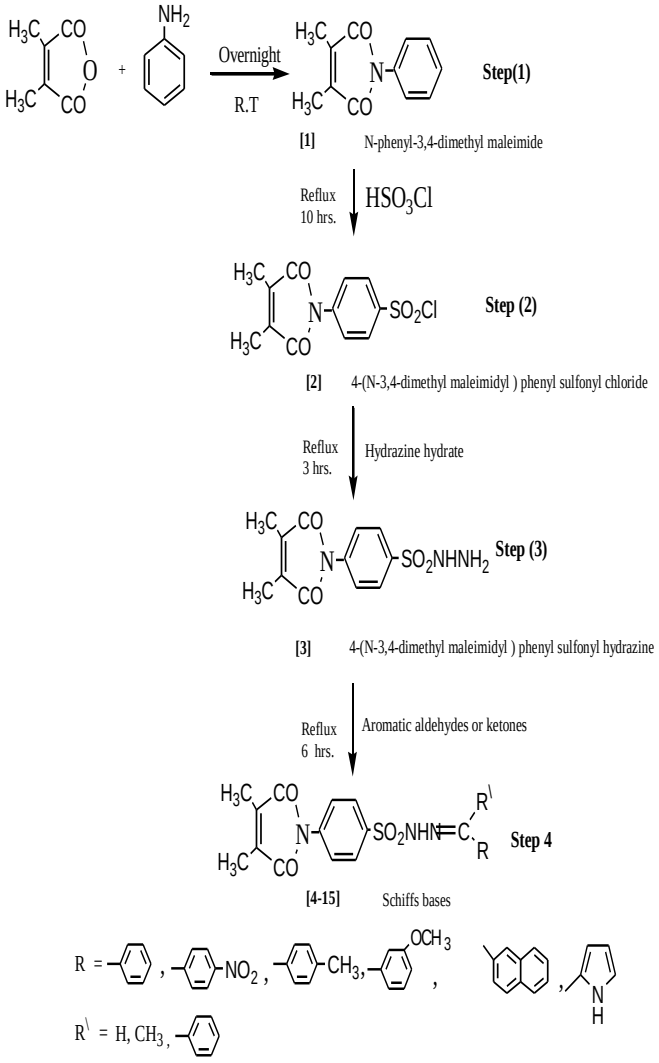
The cup plate method using nutrient agar medium was employed (18- 20) to study the antibacterial activity of the prepared Schiff bases [4-15] against staphylococcus aureus, Escherichia coli and Pseudomonas aeruginosa and dimethyl formamide was used as sample solution. Using sterilized cork borer cups were scooped out of agar medium contained in a Petri dish which was previously inoculated with the microorganisms.

The test compound solution(0.1mL) was added in the cups and the Petri dishes were subsequently incubated at 37 0C for 48 hrs. Zones of inhibition produced by each compound was measured in mm and the results are listed in Table (6).

**Results and Discussion**

Since both N- substituted 3,4-dimethyl maleimides and Schiff's bases posses biological activity and have wide spectrum of biological applications, the target of the present work has been directed toward building of new molecules containing the two active moieties dimethyl maleimide and Schiff's base respectively with expected biological activity.

Performing this target was made by multistep synthesis described in scheme (1).




**Scheme (1)**

The first step involved synthesis of 3,4-dimethyl maleimide via reaction of 3,4-dimethyl maleic anhydride with aniline under certain conditions [15]. The resulted imide [1] was introduced in chlorosulfonation reaction in the second step via reaction with chlorosulfonicacid producing compound[2] which in turn was introduced in substitution reaction with hydrazine hydrate in the third step producing dimethyl maleimidyl hydrazine derivative [3].

Finally the fourth step of this work involved reaction of compound [3] with different aldehydes and ketones producing the desirable Schiff's bases[4-15]. The strategy which we depend on in building the new Schiff's bases involved introducing sulfonyl chloride group in para position of phenyl ring attached to maleimide moiety then nucleophilic replacement of chloride with hydrazine moiety created a suitable position represented by amino group which was ready for condensation with different aldehydes and ketones producing the desirable Schiff's bases .Structures of the prepared compounds in this work were confirmed by FTIR, $^1$HNMR ,C$^{13}$NMR spectral data and C.H.N analysis.

FTIR spectrum of compound [1] showed clear absorption bands at (1704) cm$^{-1}$ and (1596) cm$^{-1}$due to υ( C=O ) imide [21] and υ(C=C ) aliphatic while FTIR spectrum of compound [2] showed appearance of two characteristic absorption bands at (1380) cm$^{-1}$and (1172) cm$^{-1}$due to asym υ($SO_2$) and sym υ($SO_2$) respectively.

FTIR spectrum of compound[3]showed appearance of NH, $NH_2$ absorption bands at (3300-3310) cm$^{-1}$ .FTIR spectra of the prepared Schiff bases [4-15] showed many clear absorption bands at (3000-3471) cm$^{-1}$,(1620-1712)cm$^{-1}$,(1525-1610) cm$^{-1}$,(1310-1380) cm$^{-1}$and (1110-1182) cm$^{-1}$ which were attributed to υ(N-H), υ(C=O )imide , υ(C=N), asym υ($SO_2$) and sym υ($SO_2$) respectively[22].FTIR spectra of compounds [9] and [12] showed absorption bands at (1203) cm$^{-1}$and (1275) cm$^{-1}$due to υ(C-O-C) ether of $OCH_3$ group.While FTIR spectra of compounds [11] and [13] showed bands at(1506-1515) cm$^{-1}$ and (1352-1404) cm$^{-1}$ due to ($NO_2$). All details of FTIR spectral data of the prepared compounds are listed at Tables (1) and (2).

On the other hand $^1$HNMR spectra for some of the prepared compounds including [1,2,3,7,8,9] showed many clear signals including clear singlet signal at δ= 2.5 ppm belong to two methyl groups which attached to imide ring , signals for aromatic protons appeared as multiplet signals at δ= (7.2-8.2) ppm in $^1$HNMR spectra of compounds [1,7,8] but appeared as doublet signals at δ= (7.0-7.9) ppm in $^1$HNMR spectra of compounds [2,3,9] and signals belong to NH amide were appeared at δ= (7.6-8.3) ppm. $^1$HNMR spectrum of compound [3] showed signal at δ= 2 ppm which was assigned to $NH_2$ group while $^1$HNMR spectrum of compound [8] showed clear signal at δ= 2.3 ppm belong to $CH_3$ group which attached to imine group and finally $^1$HNMR spectrum of compound [9] showed clear singlet signal at δ= 3.8 ppm belong to $OCH_3$ methoxy group and another signal at δ= 8.2 ppm belong to the single proton attached to imine group (-N=C-H) . All details of $^1$HNMR spectral data are listed in Table(3).

$^{13}$CNMR spectral data for compounds [1,2,3,7,8,9] were used also f or confirming their

structures. In general the spectra showed many characteristic signals including signals at (9.11-17) ppm belong to two $CH_3$ groups attached to imide ring, signals at (100-143) ppm belong to aromatic ring carbons, signals at (137.9-148) ppm belong to two vinylic (-C=C-) carbons in imide ring and signals at (164-173.2) ppm belong to two carbonyl carbons in imide ring.

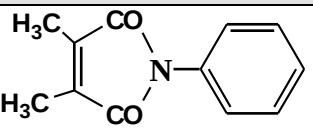
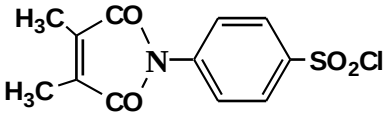
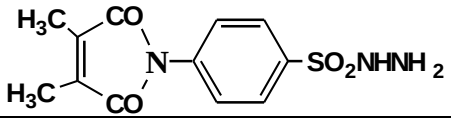
Additionally $^{13}$CNMR spectra of schiff's bases [7,8,9] showed other signals at (156-161.9) ppm which was characteristic signal for imine carbon ( ) and finally compound [9] showed clear signal at (55.6) ppm belong to ($OCH_3$) group which was attached to aromatic ring in this compound .All details of $^{13}$CNMR spectral data of the prepared compounds are listed in Table (4).

Moreover Table (5) lists C.H.N analysis for some of the prepared compounds.

The prepared Schiff's bases [4-15] were expected to possess biological activity since they were built from two biologically active components thus antibacterial activity of the prepared Schiff's bases were examined against two types strains gram positive and gram negative bacteria including *staphylococcus aureus, Escherichia coli and Pseudomonas aeruginos*a. The test results presented in Table(6) showed that most of the new Schiff's bases including (6,7,8,10,12,13,14,15) showed very high activity against staphylococcus aureus while compounds (4,5,9,11) showed high activity against this bacteria. The results showed also that the new compounds are highly active against pseudomonas aeruginosa except compounds (7,11) which are moderately active.

Finally the results indicated high activity of the prepared Schiff's bases against Escherichia Coli except compound(10) which showed moderate activity and compound(7) which was inactive. As a final conclusion it is interesting to mention that incorporation of 3,4- dimethyl maleimide moiety in Schiff's bases molecules exhibit high biological (antibacterial) activity and this is fitted with our expections.

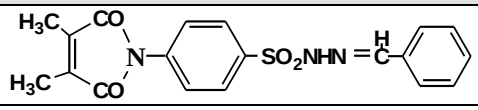
**Table (1) Melting points , percent yields and FTIR data of compounds [1-3]**

| No. | Compound Structure | Yield % | Melting Point C$^0$ | Major FTIR Absorptions Cm$^{-1}$ * | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | υ(N-H) | υc=o | υc=c | υ($SO_2$) Asym | υ($SO_2$) Sym |
| 1 |  | 77 | 90-91 | ---- | 1704 | 1596 | ---- | ---- |
| 2 |  | 81 | 165-166 | ---- | 1720 | 1589 | 1380 | 1172 |
| 3 |  | 75 | 180-182 | 3310 | 1627 | 1527 | 1365 | 1164 |

*** As KBr disc**

**Table (2) some physical properties and FTIR data of the prepared Schiff's bases**

*** As KBr disc**

| Comp. No. | Compound Structure | Yield % | Melting Point C$^0$ | * Major FTIR Absorptions Cm$^{-1}$ C=N C=O | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | | υ(N-H) | υ | υ | υ($SO_2$) Asym | υ($SO_2$) Sym |
| 4 |  | 81 | 110-112 | 3420 | 1674 | 1610 | 1363 | 1182 |

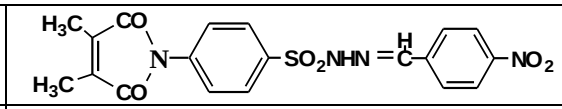
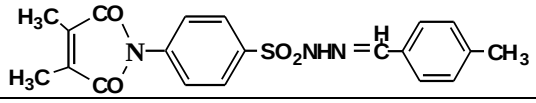
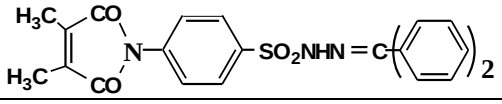
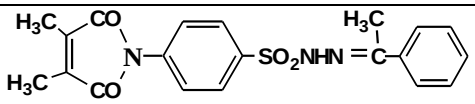
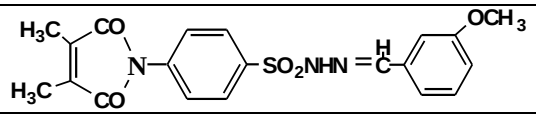
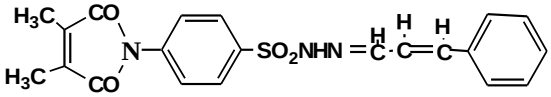
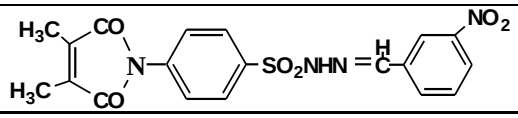
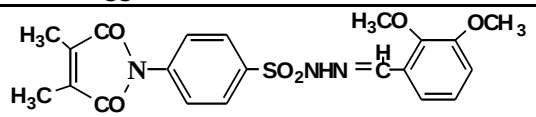
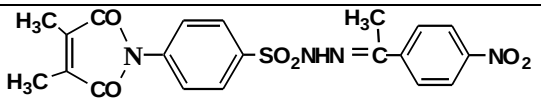
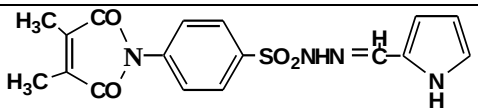
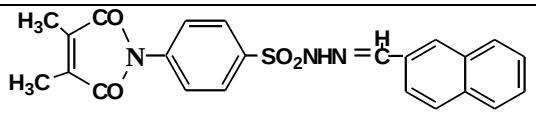
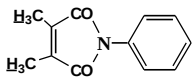
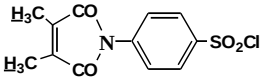
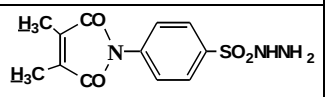
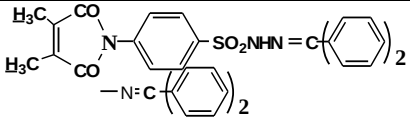
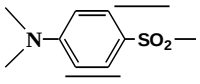
| | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| 5 |  | **77** | **141-143** | **3400** | **1620** | **1525** | **1342** | **1110** |
| 6 |  | **84** | **125-126** | **3410** | **1683** | **1548** | **1371** | **1158** |
| 7 |  | **75** | **162-163** | **3456** | **1674** | **1590** | **1319** | **1180** |
| 8 |  | **85** | **115-116** | **3471** | **1620** | **1566** | **1357** | **1180** |
| 9 |  | **88** | **131-133** | **3224** | **1658** | **1590** | **1326** | **1157** |
| 10 |  | **78** | **139-140** | **3363** | **1712** | **1596** | **1310** | **1180** |
| 11 |  | **71** | **157-158** | **3390** | **1670** | **1545** | **1380** | **1170** |
| 12 |  | **82** | **120-122** | **3380** | **1650** | **1560** | **1350** | **1175** |
| 13 |  | **80** | **202-204** | **3394** | **1658** | **1589** | **1342** | **1172** |
| 14 |  | **75** | **153-154** | **3217** | **1628** | **1545** | **1332** | **1134** |
| 15 |  | **72** | **160Dec.** | **3000** | **1620** | **1581** | **1319** | **1180** |

Table (3) $^{1}$H-NMR spectral data for some of the prepared compounds

| Comp. No. | $^{1}$H-NMR spectral data |
|---|---|
| 1 | δ =2.5 (s) ppm, 6H of 2$CH_3$ ,<br>δ = 7.8-8 (m) ppm 5H aromatic  |
| 2 | δ =2.5(s) ppm, 6H of 2$CH_3$ ,  <br>δ = δ7.3(d) ppm 2H aromatic , δ= 7.7 (d) ppm , 2H aromatic |
| 3 | δ=2 (s)ppm,$NH_2$ ,δ= 2.5(s) ppm, 6 Hof 2$CH_3$,  <br>, δ =7-8(d) ppm, 2H aromatic, δ=7.9(d) ppm , 2H aromatic |
| 7 | δ =2.5(s) ppm , 6H of 2 $CH_3$<br>δ= (7.27-7.4) (m)ppm , 10 H aromatic  <br>δ= (7.4-7.5)(m) ppm,4H aromatic<br>δ = 7.6(s) ppm , NH amide  |

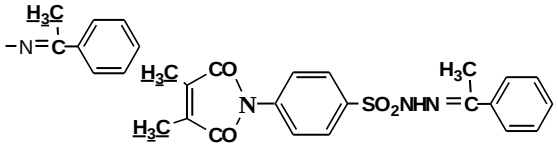
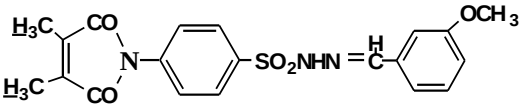
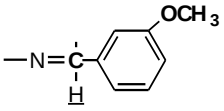
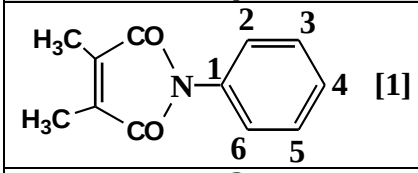
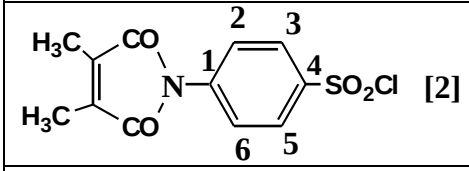
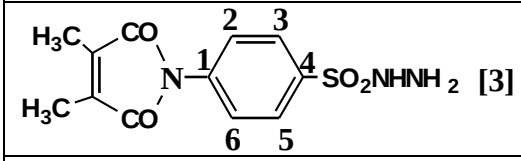
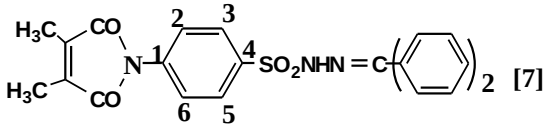
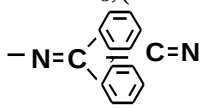
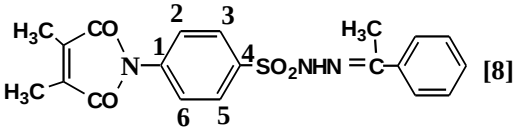
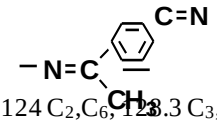
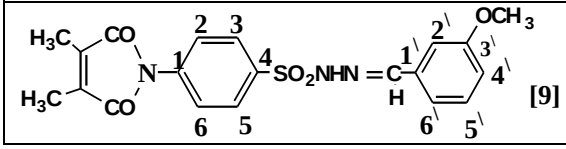
| | |
|---|---|
| 8 | δ= 2.3(s) ppm , 3H of $CH_3$<br>δ = 2.5 (s)ppm , 6H of 2 $CH_3$<br>  <br>δ = (8.1-8.2) (m)ppm, 9H aromatic , δ= 8.3(s) ppm , NH amide |
| 9 | δ= 2.5(s) ppm , 6H of 2 $CH_3$<br>δ =3.8(s) ppm ,3H of $OCH_3$,<br>  <br>δ = (7-7.4)(d) ppm 4 H aromatic , δ= 8(s) ppm, NH amide<br>  <br>δ= 8.2(s) ppm,1H |

Table (4) C13NMR data for some of the prepared compounds

| Compound structure | $C^{13}$NMR data (ppm) |
|---|---|
|  [1] | 11.29 ppm 2$CH_3$ , 129.07 ppm $C_4$ , 130.07ppm $C_3$,$C_5$<br>131.5 ppm $C_2$ and $C_6$, 134.7 ppm $C_1$, 139.66 ppm two vinylic carbons,<br>173.2 ppm two carbonyl carbons |
|  [2] | 9.11 pm 2$CH_3$,126.3 ppm $C_2$,$C_6$ , 132.5 ppm $C_3$,$C_5$, 137.5 ppm $C_1$,$C_4$,<br>147.3 ppm two vinylic carbons , 170.97 ppm two carbonyl carbons |
|  [3] | 9.6 2$CH_3$, 127 $C_2$,$C_6$, 131 $C_3$,$C_5$ , 135 $C_1$,$C_4$, 144.3 two vinylic carbons,<br>170.1 ppm two carbonyl carbons |
|  [7] | 16.9 2$CH_3$, (127.9-129.5) 12 aromatic ring carbons<br>  <br>130 $C_2$,$C_6$, 130.4 $C_3$,$C_5$ ,135$C_1$, 135.5 $C_4$, 137.9 two vinylic carbons ,<br>159.5 , 170 two carbonyl carbons |
|  [8] | 15.55 3 $CH_3$ groups, (100-101) 6 aromatic ring carbons<br>  <br>124 $C_2$,$C_6$, 128.3 $C_3$,$C_5$, 143 $C_1$,$C_4$, 148 two vinylic carbons , 156 ,164<br>two carbonyl carbons. |
|  [9] | 16.2, 17 2$CH_3$, 55.6 $OCH_3$, 112C\5,C\6, 116.2 $C_2$,$C_6$, 118.5$C_3$,$C_5$, 120.4<br>C\4, 121.7 C\2,130 C\1, 136.1 $C_1$, 143 $C_4$,146 two vinylic carbons, 159.9<br>C\3, 161.9 , , 171.2 two carbonyl carbons. |

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**Table(5) C.H.N analysis of the prepared compounds**

| Comp. No. | Calculated | | | Found | | |
|---|---|---|---|---|---|---|
| | %C | %H | %N | %C | %H | %N |
| 1 | 73.09 | 5.58 | 7.10 | 72.93 | 5.49 | 6.98 |
| 2 | 48.08 | 3.33 | 4.67 | 48.25 | 3.53 | 4.80 |
| 3 | 48.81 | 4.40 | 14.23 | 49.02 | 4.29 | 14.30 |
| 4 | 59.53 | 4.43 | 10.96 | 59.37 | 4.44 | 10.88 |
| 5 | 53.27 | 3.73 | 13.08 | 53.04 | 3.60 | 13.19 |
| 7 | 65.35 | 4.57 | 9.15 | 65.31 | 4.46 | 9.00 |
| 8 | 60.45 | 4.78 | 10.57 | 60.27 | 4.71 | 10.39 |
| 9 | 58.11 | 4.60 | 10.16 | 58.20 | 4.74 | 10.32 |
| 13 | 54.29 | 4.07 | 12.66 | 54.40 | 4.17 | 12.54 |

**Table (6) Antibacterial activity of Schiff 's bases (4-15)**

| Comp. No. | Gram positive bacterria | Gram negative bacteria | |
|---|---|---|---|
| | *S.aureus* | *P.aeruginosa* | *E.Coli* |
| 4 | +++ | +++ | +++ |
| 5 | +++ | +++ | +++ |
| 6 | ++++ | +++ | +++ |
| 7 | ++++ | ++ | - |
| 8 | ++++ | +++ | +++ |
| 9 | +++ | +++ | +++ |
| 10 | ++++ | +++ | ++ |
| 11 | +++ | ++ | ++ |
| 12 | ++++ | +++ | +++ |
| 13 | ++++ | +++ | +++ |
| 14 | ++++ | +++ | +++ |
| 15 | ++++ | +++ | |

**Key to symbols : Inhibition zone < 6= - Inactive**
**Slightly active 6-9 = +**
**Moderately active 9-12= ++**
**Highly active 13-16= +++**
**Very high activity > 17= ++++**

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# تحضير , تشخيص ودراسة الفعالية البايولوجية لقواعد شيف جديدة حاوية على المكونة 4,3- ثنائي مثيل مالي ايمايد

**احلام معروف العزاوي**

**الخلاصة**

تم في هذا البحث تحضير سلسلة من قواعد شيف الجديدة الحاوية في تركيبها على المكونة 4,3-ثنائي مثيل مالي ايمايد وذلك بإتبا ع طريقة التحضير المتعدد الخطوات حيث تم في الخطوة الأولى تفاعل الانيلين مع 4,3-ثنائي مثيل انهيدريد الماليك لتكوين المركب N-فنيل-4,3-ثنائي مثيل مالي ايمايد وهذا بدوره تم مفاعلته في الخطوة الثانية مع حامض كلوروسلفونيك للحصول على المركب 4-(N-4,3-ثنائي مثيل مالي ايميديل) فذيل كلوريد السلفونيل الذي تمت معاملته مع هيدرازين المائي في الخطوة الثالثة لانتاج المركب 4-(N-4,3-ثنائي مثيل مالي ايميديل) فنيل سلفونيل هيدرازين و هذا الاخير عند تكاثفه مع الديهايدات و كيتونات اروماتية مختلفة اسفر عن تكوين قواعد شيف المطلوبة .تم اثبات تراكيب المركبات المحضرة بالاعتماد على مطيافية الاشعة تحت الحمراء FTIR والرنين النووي المغناطيسي1HNMR و C13NMR بالاضافة الى تحليل العناصر. كذلك تمت دراسة الفعالية البايولوجية لقواعد شيف المحضرة ضد ثلاثة انواع من البكتريا هي على التوالي ستافيلوكوكاس اوريس, اشريشيا كولي و بسيدوموناس اوريجينوزا وقد اوضحت نتائج الدراسة بان معظم قواعد شيف المحضرة ذات فعالية بايولوجية عالية ضد انواع البكتريا المذكورة.