

SYNTHESIS AND SPECTROSCOPIC CHARACTERIZATION OF A NEW SERIES OF BENZO[E][1,3]OXAZEPINE COMPOUNDS FROM SCHIFF BASES

Hashim Jalal Azeez ^{a,*}, Kali Mohammed Qadir ^a

^a Dept. of Chemistry, College of Education, University of Salahaddin - Hawler, Erbil-Kurdistan, Iraq - hashim1961@ymail.com

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ABSTRACT:

The present work includes synthesis, characterization and investigation the biological activity of a new series of benzo [e][1, 3]oxazepine compounds which were prepared from Schiff bases. The synthetic routes have been divided into two steps as follows: The first step includes the synthesis of Schiff bases, by treatment of 4-amino diphenyl amine and different substituted benzaldehydes in absolute ethanol using glacial acetic acid as catalyst. The second step of this work is the synthesis of a series benzo [e][1, 3] oxazepine, by the reaction of the synthesized Schiff bases with phthalic anhydride in dry benzene. The structures of the synthesized products were confirmed on the basis of (UV-Visible, FT-IR, ¹H-NMR, ¹³C-NMR spectroscopy. Some of the synthesized compounds were evaluated for antibacterial activity, against two types of bacteria, *Staphylococcus aureus* (Gram positive) and *Pseudomonas aeruginosa* (Gram negative). The results showed the high sensitivity of the synthesized compounds to both types of bacteria.

KEYWORDS: Schiff Base; Benzo [e][1, 3] Oxazepine; Antimicrobial Activity.

1. INTRODCUTION

The Schiff bases play a vital role and find its use in analytical chemistry, agriculture, dyes and polymer industries, besides their utility as model systems in the field of bio-inorganic chemistry (Ren et al., 2006), biological activities and industrial application (Wang et al., 2006), Seven membered heterocycle containing two hetero atoms are oxygen in position (1) and nitrogen in position (3) in addition of five carbon atoms. 1, 3-oxazepines are prepared by condensation of Schiff bases with anhydrides (maleic anhydride, phthalic anhydride, nitro phthalic anhydride, succinic anhydride), to give corresponding cycloaddition products. A cycloaddition reaction most commonly involves two molecules reacting to form two new sigma bonds between the end atoms of their pi systems resulting in the formation of a ring.

The reaction of these anhydrides with Schiff bases is classified as a 5+2 =7, 5-atom component plus 2-atom component leading to 7-membered cyclic ring (Hanoon, 2011). Benzoxazepines are a seven-membered unsaturated heterocyclic compounds that contains two hetero atoms (oxygen and nitrogen) (Ahmed and Al-Hashimi, 2016). Oxazepine derivatives are found to exhibit a vast variety of biological activities like antibacterial (Hikmet et al., 2013), antifungal (Serrano-wu et al., 2002), hypnotic muscle relaxant (Abdelhafez and Abdelwahab, 2008), antagonistic (Hallinan et al., 1996), anti-inflammatory (Kubota et al., 2011), telomerase inhibitors (Xin-Hua et al., 2013) and antiepileptic (Kiran et al., 2003).

2. MATERIALS AND METHODS

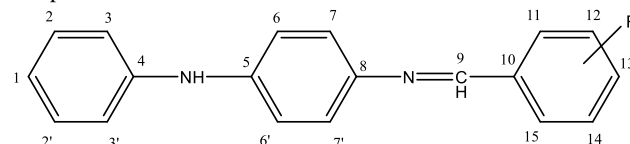
2.1 Experimental notes

Melting points were determined using an Electrothermal melting point apparatus. IR spectra were recorded on IR Affinity-1 Spectrophotometer, using KBr disc. ¹H-NMR, and

¹³C-NMR spectra were recorded on a Bruker (300 & 400MHz) with TMS as internal reference, using DMSO as solvent, in Jordan University of science and technology, Irbid.

2.2 Synthesis of *N*¹ - (substituted benzylidene)-*N*⁴-phenylbenzene-1,4-diamine (2 a-j) (AL-jamali, 2015)

In round bottom flask (1.84g, 0.01 mole) of 4-aminodiphenyl amine (1) in (50 mL) of absolute ethanol, (0.01 mole) of appropriate aromatic aldehyde with some drops of glacial acetic acid was refluxed for (3-7 hrs). The reaction mixture was then allowed to cool to room temperature and solid product was filtered and recrystallized from ethanol to give colored compounds.



2.2.1 Synthesis of *N*¹ - (4-Bromobenzylidene)-*N*⁴-phenylbenzene-1,4-diamine (2d): (C₁₉H₁₅N₂Br), m.p. (141-143 °C), yield (91 %); ¹H-NMR: 7.7-8.4 (m, 13H, aromatic ring), 8.7 (s, 1H, -NH), 9 (s, 1H, Schiff base proton-CH=N-). ¹³C-NMR: 116.52: C₁, 116.67: C_{3,3'}, 119.65: C_{6,6'}, 122.27: C_{7,7'}, 123.89: C₁₃, 128.92: C_{2,2'}, 129.71: C_{11,15}, 131.49: C_{12,14}, 135.39: C₁₀, 142.11: C₅, 142.17: C₄, 142.73: C₈, 155.18: (CH=N) C₉. ¹³C-DEPT: 116.5 C₁; 116.7: C_{3,3'}, 119.7: C_{6,6'}, 122.3: C_{7,7'}, 128.9: C_{2,2'}, 129.7: C_{11,15}, 131.4: C_{12,14}, 155.2: (CH=N) C₉.

* Corresponding author

2.2.2 Synthesis of N^1 - (N,N-dimethylbenzylidene)- N^4 -phenylbenzene-1,4-diamine (2f): ($C_{21}H_{21}N_3$), m.p. (125-127 °C), yield (81 %); 1H -NMR: 4.4 (s, 6H, 2CH₃), 6.54-7.8 (m, 13H, aromatic ring), 8.2 (s, 1H, -NH), 8.43(s, 1H, Schiff base proton-CH=N-). ^{13}C -NMR: 45.2: 2CH₃,111.09: C12,14,115.93: C1,117.12: C3,3',118.97: C6,6',121.49: C7,7', 123.88: C10, 128.76: 2,2',129.40 :C11,15,140.52: C5,143.19: C4,143.89: C8,151.59: C13,156.48: (CH=N)C9. ^{13}C -DEPT: 116.6;C1, 116.7; C3,3',119.5; C6,6',122.1; C7,7',128; C12,14,128.5;C11,15,128.9; C2,2', 130.5:C13 ,156.6:(CH=N) C9.

2.2.3 Synthesis of N^1 - (benzylidene)- N^4 -phenylbenzene-1,4-diamine (2g): ($C_{19}H_{16}N_2$), m.p. (99-100 °C), yield (82 %); 1H -NMR: 6.83-7.39 (m, 14H, aromatic ring), 8.33 (s, 1H, -NH), 8.66 (s, 1H, Schiff base proton-CH=N-) ^{13}C -NMR: 116.55: C1,116.72; C3,3',119.53; C6,6',122.11;C7,7',127.94; C12,14,128.44; C11,15,128.91; C2,2',130.52; C13,136.18; C10,141.86; C5,142.63; C4,142.90; C8,156.55; (CH=N) C9.

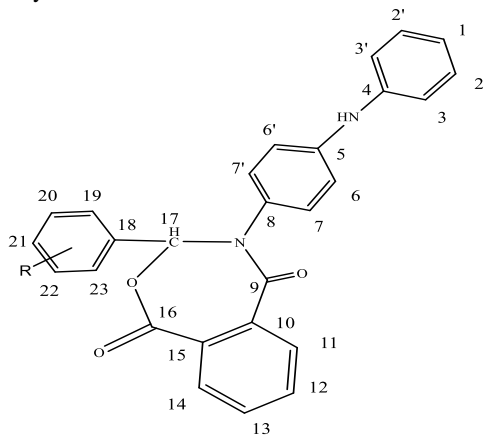
2.2.4 Synthesis of N^1 - (2-hydroxybenzylidene)- N^4 -phenylbenzene-1,4-diamine (2h): ($C_{19}H_{16}N_2O$), m.p. (103-105 °C), yield (84 %); 1H -NMR: 6.85-7.8 (m, 13H, aromatic ring), 8.93(s, 1H, -NH), 9.1(s, 1H, Schiff base proton-CH=N), ^{13}C -NMR: 116.03: C12,,116.29; C1,116.87; C10, 118.57; C3,3',119.09; C6,6',119.82; C14,122.11; C7,7',128.84; C2,2',131.71; C15,132.03; C13,138.79; C5,142.37; C4,142.60; C8,159.13; (CH=N) C9,159.73; C11.

2.2.5 Synthesis of N^1 - (2-fluorobenzylidene)- N^4 -phenylbenzene-1,4-diamine (2i): m.p. (91-93 °C), yield (88 %); 1H -NMR: 6.83-7.8 (m, 13H, aromatic ring), 8.28 (s, 1H, -NH), 8.59 (s, 1H, Schiff base proton-CH=N-)

2.2.6 Synthesis of N^1 - (3-nitrobenzylidene)- N^4 -phenylbenzene-1,4-diamine (2j): ($C_{19}H_{15}N_3O_2$), m.p. (112-114 °C), yield (95 %); 1H -NMR: 6.84-8.4 (m, 13H, aromatic ring), 8.69 (s, 1H, -NH), 8.82(s, 1H, Schiff base proton-CH=N-). 116.12; C1,116.77; C3,3',119.70; C6,6',121.65; C7,7', 122.44; C13,124.41; C11,128.77; C2,2',129.91; C14,133.68; C10,137.68; C15m,141.21; C5,142.37; C4,142.55; C8,147.72; C12,153.79; (CH=N) C9.

2.3 Synthesis of (Oxazepine derivatives):3-sustituted phenyl-4-(4-(phenylamino)phenyl)-3,4-dihydro-benzo[e][1,3]oxazepine-1,5-dione (3 a-j), (Alexander *et al.*, 2016).

Mixture of Schiff base (2mmol) and phthalic anhydride (2mmol,0.0296gm) in (20mL) dry benzene was heated for (5-6 hrs) in water bath at (85°C), the precipitate was filtered and recrystallized from toluene



2.3.1 3-(4-bromophenyl)-4-(4-(phenylamino)phenyl)-3,4-dihydro-benzo[e][1,3]oxazepine-1,5-dione (3d):

($C_{27}H_{19}N_2O_3Br$), m.p. (198-200 °C), yield (96 %); 1H -NMR: 6.75-7.87 (m, 17H, aromatic rings, phthalic anhydride), 8.06 (s, 1H, -NH),10.17 (s, 1H, -C-H proton). ^{13}C -NMR:115.28; C17,117.37; C1,118.53; C3,3',120.38; C6,6',127.07; C21,127.05C7,7', 128.11; C11,128.73; C8,128.77; C19,23,128.9; C2,2',130.34; C14,15,131.17; C10,132; C20,22,132.48; C12,13,138.52;C5,143.43; C18,142; C4,167.26; C16C=O,168.23; C9C=O. ^{13}C -DEPT: 115.3; C17, 117.4, C1, 118.5; C3,3',120.4; C6,6',127.4; C7,7',128.1; C11,128.7; C19,23,128.8; C2,2',129; C20,22,130.02; C12,13.

2.3.2 3-(N,N-dimethylphenyl)-4-(4-(phenylamino)phenyl)-3, 4-dihydro-benzo[e][1,3]oxazepine-1,5-dione (3f): ($C_{29}H_{25}N_3O_3$), m.p. (168-170 °C), yield (96 %); 1H -NMR:3.55 (s, 6H, 2CH₃), 6.68-8.29 (m, 17H, aromatic rings) 8.56 (s, 1H, -NH), 9.67 (s, 1H, -C-H proton).

2.3.3 3-(phenyl)-4-(4-(phenylamino)phenyl)-3,4-dihydro-benzo[e][1,3]oxazepine-1,5-dione (3g): ($C_{27}H_{20}N_2O_3$), m.p. (175-176 °C), yield (60 %); 1H -NMR6.75-7.87 (m, 18H, aromatic rings), 8.06 (s, 1H, -NH),10.17 (s, 1H, -C-H proton): ^{13}C -NMR: 115.36; C17,117.35; C1,117.44; C3,3', 118.59; C6,6',120.45; C7,7',127.41; C21,128.77; C19,23,129.44; C11,129.83; C20,22,8,129.86; C2,2',129.9; C14,15,130.35; C10,131.20; C13,132; C12,133; C5,138.77; C4,143.86; C18,167.23; C16C=O,168.23; C9C=O.

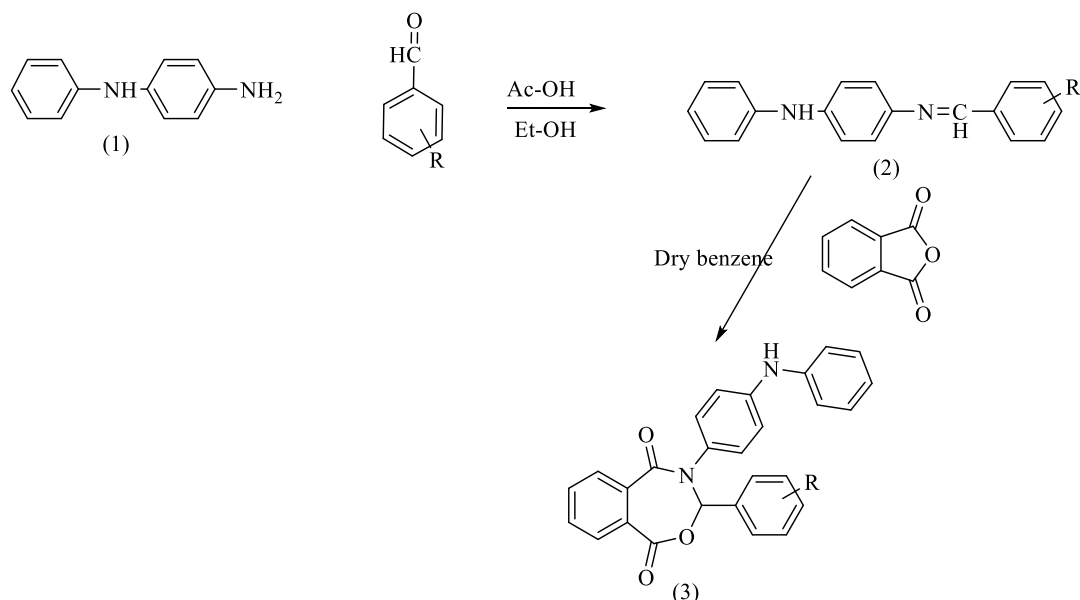
2.3.4 3-(2-hydroxyphenyl)-4-(4-(phenylamino)phenyl)-3,4-dihydro-benzo[e][1,3]oxazepine-1,5-dione (3h) : ($C_{27}H_{20}N_2O_4$), m.p. (128-130 °C), yield (69 %); 1H -NMR :6.75-8.4 (m, 17H, aromatic rings), 8.92 (s, 1H, -NH),10.17 (s, 1H, -C-H proton) 13.48 (br,1H, -OH). ^{13}C -NMR: 115.24;C17,115.99; C20,116.25; C1,116.83; C3,3',117.32; C6,6',118.55; C22,119.04;C7,7', 119.80;C11,120.34; C21,122.07; C23,127.31; C8,128.69; C18,128.80; C2,2',128.98; C14,15,131.67; C10,132.01; C12,13,142.56; C5,159.11; C4,159.67; C19,166.5; C16C=O,167.1C9C=O. ^{13}C -DEPT: 115.23; C17,115.98; C20,117.31; C1,116.85; C3,3',117.34; C6,6',118.58; C22,119.06; C7,7',119.82; C11, 120.36; C21,122.08; C23,128.82; C2,2',128.99; C14,132.01; C12,13.

2.3.5 3-(2-fluorophenyl)-4-(4-(phenylamino)phenyl)-3,4-dihydro-benzo[e][1,3]oxazepine-1,5-dione (3i) : ($C_{27}H_{19}N_2O_3F$), m.p. (120-122 °C), yield (53 %); 6.75-8.72 (m, 17H, aromatic rings), 8.86 (s, 1H, -NH),10.18 (s, 1H, -C-H proton). ^{13}C -NMR: 115.43;C17,115.99: C1,117.52;C3,3',21, 117.99;C6,6',118.67: C19,120.53: C7,7',127.50: C11,128.85: C8,128.96C22,129.15: C2,2',129.84: C14,15,130.40: C10,131.28:C13,131.99: C23,132.23:C12,132.70: C5 , 138.57: C4,138.70: C18,143.84:C20, 166.51:C16,C=O,167.32:C9,C=O.

3. RESULTS AND DISCUSSION

3.1 Synthesis and characterization

In the present work we described the synthesise and characterization of some new oxazepine derivatives along with biological evaluation against two types of bacteria *Escherichia coli* gram negative and *Staphylococcus aureus* gram positive. The preparation processes as outlined in Scheme (1).



Where R a=4-NO₂, b= 4-F, c= 4-Cl,d= 4-Br, e= 4-OCH₃, f= 4-(CH₃)₂ , g= H, h = 2-OH, i = 2-F, j= 3-NO₂

Scheme 1. Oxazepin(3) synthesis from Schiff bases (2)

Table 1. Assignment of characteristic frequencies (cm⁻¹) of IR spectral data for the synthesized Schiff bases (2 a-j)

Comp. No.	R	C-H str. Aromatic	C-H str. Schiff base	CH=N str.	C=C str. Aromatic	-NO ₂	-NH str.
2a	4-NO ₂	3066	2882	1626	1597	1512,	3414
2b	4-F	3050	2860	1624	1596	-----	3414
2c	4-Cl	3025	2876	1620	1593	-----	3414
2d	4-Br	3028	2872	1622	1595	-----	3412
2e	4-OCH ₃	3070	2850	1621	1598	-----	3372
2f	4-N(CH ₃) ₂	3030	2881	1597	1577	-----	3232
2g	-H	3032	2877	1622	1587	-----	3414
2h	2-OH	3049	2885	1622	1597	-----	3346
2i	2-F	3024	2878	1623	1602	-----	3397
2j	3-NO ₂	3032	2882	1627	1589	1524, 1350	3383

Table 2. Assignment of characteristic frequencies (cm⁻¹) of IR spectral data for the synthesized 3a-j

Comp. No.	R	C-H str. Aromatic	C-H str. of oxazepine ring	C=O str. C-O str. Lacton	C=O str Lactam	C=C str. Aromatic	-NH str.
3a	4-NO ₂	3034	2940	1722.43;1240.23	1647.21	1599	3377
3b	4-F	3060	2893	1705.07;1244.09	1651.07	1597.06	3385
3c	4-Cl	3054	2890	1703.14;1282.66	1691.27	1560.41	3387
3d	4-Br	3070	2899	1722.43;1242.16	1695.43	1602	3377
3e	4-OCH ₃	3038	2870	1720.50;1244.09	1705	1516	3310
3f	4-N(CH ₃) ₂	3053	2856	1716.65;1243.04	1653	1595	3315
3g	-H	3032	2887	1720.50;1242.16	1701.22	1598	3379
3h	2-OH	3026	2879.7	1722.43;1244.09	1699.29	1597	3311
3i	2-F	3011	2882	1701.22;1280.73	1683.86	1587.42	3385
3j	3-NO ₂	3053	2937	1722.43;1242.16	1647.21	1600.92	3377

The substrate 4-aminodiphenylamine (1) reacted with different aromatic benzaldehydes in absolute ethanol to, was cyclized when treated with phthalic anhydride to give a

series of oxazepines(3).The formations of new oxazepines (3a-j) were identified on the basis of their spectral methods: (UV-Visible, FT-IR, ¹H-NMR, and ¹³C-NMR),Table (2).

The general feature of the IR spectra of oxazepines (3 a-j) which has been shown in Table (2) consist in the presence of strong sharp bands at (1701-1722) cm^{-1} and the appearance of the strong absorption bands at (1592-1705) were due to the stretching vibration of the (C=O) lactone and lactam groups respectively of the oxazepine compounds that is considered as an evidence for the formation of described products. The disappearance of the (C=N) band for Schiff bases in the range of the (1627-1597) cm^{-1} in all 1, 3-oxazepines is also a strong evidence for the occurrence of oxazepine compounds (Aljamali, 2015).

3.2 Biological activity

Schiff bases and oxazepines were screened to anti-bacterial activities against two types of bacteria Gram-positive (*Staphylococcus aureus*) and Gram negative bacteria (*Escherichia coli*) as KBr discs in a sterilized Muller Hinton agar medium. The prepared discs placed on the surface of the cultured media was incubated at 37 °C for 24 hours. The Zone of inhibition produced by each compound was measured in mm; most of them recorded a high order of anti-bacterial activity against Gram negative *S-aureus* Gram +ve bacteria) [2c: 35, 2e: 33, 2g:22,2i:33,3c: 40, 3d: 30, 3e: 36, 3i: 39,3g:39mm] and *P. aeruginosa* Gram -ve bacteria [2c: 33, 2e: 40, 2g:40,2i:37,3c: 35, 3d: 22, 3e: 30, 3i: 40,3g:40mm]. The results showed that the most of compounds (Schiff bases and oxazepines) showed highly effective against two types of bacteria, while the compounds (3g, 4 d) showed moderately active, Table (3).

Table 3. The antibacterial activity of some of the synthesized Schiff base and Oxazepines with inhibition zone diameters in (mm) scale against two strain of bacteria.

Compounds	Zone of inhibition in (mm)	
	<i>S-aureus</i> Gram +ve bacteria	<i>P. aeruginosa</i> Gram -ve bacteria
2 c	35	33
2 e	33	40
2 g	22	40
2 i	33	37
3 c	40	35
3 d	30	22
3 e	36	30
3 i	39	40
3 g	39	40

4. CONCLUSION

The prepared new Schiff base and heterocyclic derivatives containing oxazepine ring are biological active against both types of bacteria *Staphylococcus aureus* and *Pseudomonas aeruginosa*. The results of biological activity for synthesized compounds were showed good effect against *Staphylococcus aureus* (Gram +ve) and *Pseudomonas aeruginosa* (Gram -ve). All compounds were found to be stable at room temperature. An electron-withdrawing group on benzaldehydes lead to formation of Schiff bases and oxazepine in higher yields than electron donating group because of the increasing the of positivity or electrophilicity of the carbon of carbonyl group in substituted benzaldehydes.

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APPENDIX

Some related figures.

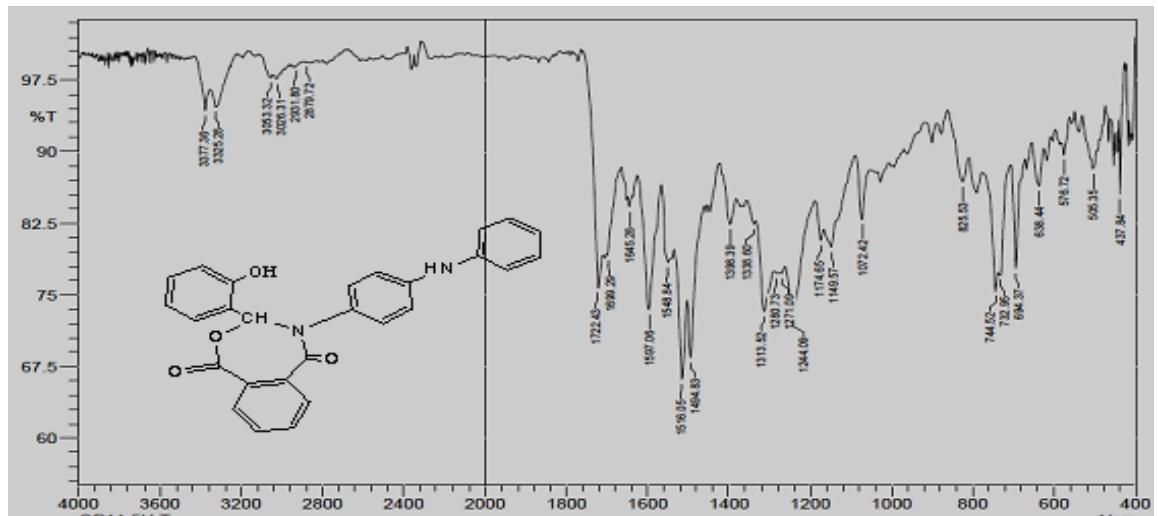


Figure 1. IR spectrum of oxazepine (3h)

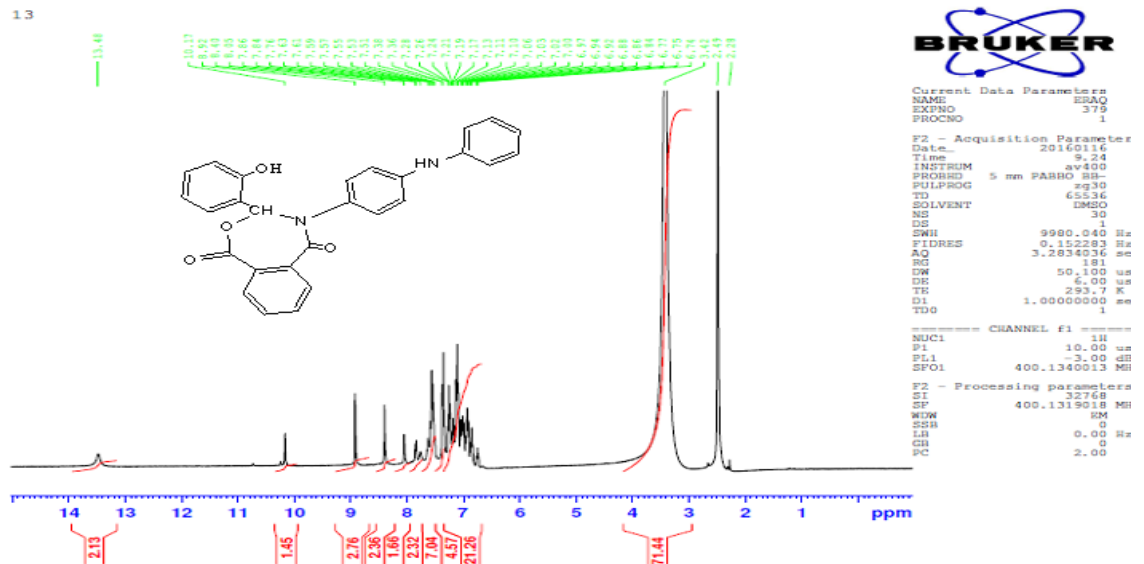


Figure 2. ¹H-NMR spectrum of oxazepine (3h)

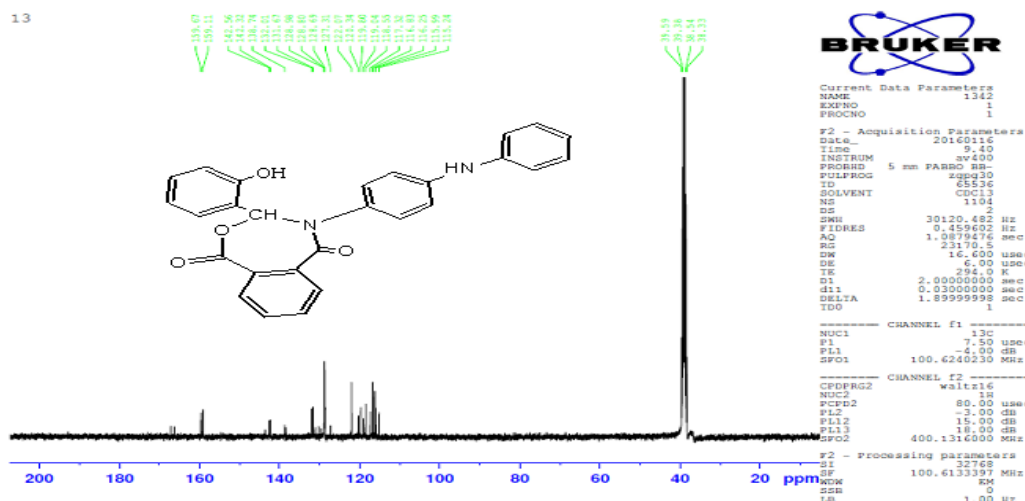


Figure 3. ¹³C-NMR spectrum of oxazepine (3h)

ئاماده كردن و دەستنيشانكردي شه به نگی زنجيره يه كى نوئ له ئاوئته كانى بئنزؤ [e] [١,٣] ئوكسازيبين له ئاوئته كانى تفتى شيف

كورتيا ليكولينى:

له م توئزبنه وه يه دا ئاماده كردن و دەستنيشانكردي شه به نگی بۇ زنجيره ي بئنزؤ [e] [١,٣] ئوكسازيبينى نوئ و شيوگى بيكهاته ي و چالاکى بايه لؤجى ده خه ينه روو له ئاوئته كانى تفتى شيف ، پرؤسه ي ئاماده كرنه كه دابه شكرا بؤ دوو به شى سه ره كى به م شيوه يه ي خواره وه: به شى يه كه م بريته له ئاماده كردنى هه ندئ له ئاوئته ي تفتى شيف (2 a-j) له ٤ - ئه مينو داى فه نيل ئه مينو داى كارليكردينان له گه ل چه ند به نزه لديهايدى له برى خراو به هؤى به كار هينانى ئيسانؤلى په تى وه كو توئنه ر به بوونى ترشى سر كيكى به فرى وه ك كاراى ياريدده ر. به شى دووه مى ليكؤلينه وه كه بريته بوو له ئاماده كردنى زنجيره ي نوئ له بئنزؤ [e] [١,٣] ئوكسازيبينى (3 a-j) به هؤى كارليكى نيوان ئيميني ئاماده كراو (2 a-j) له گه ل ئاوئته ي فساليك ئه نه دريد له ناو به نزيى وشكدا. شيوگى كيمياوى ماده به ره م هاتوه كان دياريكرا ن به به كار هينانى ريگاي شه به نكه كان وه ك شه به نگی ژير سوور (FT-IR) وشه به نگی سه رووى وه نه وشه يى و بينراو هه وروه ها شه به نگی له رينه وه ي ناوكى موگناتيسى ($^{13}\text{C-NMR}$, $^1\text{H-NMR}$, UV-Visible). هه ندئ له ئاوئته به ره م هاتوه كان تافيكرا نه وه له روى دژه به كتر يه وه دژى دوو جوړ له به كتر يه گرام نيگه تيف (*P. aeruginosa*) له گه ل گرام پؤزه تيف (*S. aureus*) وه له ئه نجامه كان هه ستيارى به رزى ئاوئته ئاماده كراوه كان ده رده خه ن دژى هه ر دوو جوړى به كتر يه

التحضير و التشخيص الطيفي لسلسلة جديدة من المركبات بنزو (e) [3و1] او كسازيبين من قواعد شف

خلاصة البحث:

يتضمن البحث تحضير و التشخيص الطيفي ودراسة الفعالية البايولوجية لسلسلة جديدة من المركبات بنزو (e) [3و1] او كسازيبين من قواعد شف. ان طريقة العمل تقسم الى خطوتان: الخطوة الاولى تفاعل 4-امينو ثنائى فينيل امين مع مختلف بنزاليهايدات المختلفة في الايثانول المطلق باستعمال حامض الخليك الثلجي كعامل مساعد. أما الخطوة الثانية هي تحضير لسلسلة جديدة من المركبات بنز (e) [1,3] او كسازيبين بواسطة تفاعل قواعد الشف المحضرة مع انه دريد الفعاليك في البنزين الجاف. تم التأكد على تركيب المركبات المحضرة على اساس اطيف $^{13}\text{C-NMR}$, $^1\text{H-NMR}$, FT-IR, UV-Visible. تم دراسة تأثير بعض المركبات المحضرة ضد نوعي من البكتريا (*Pseudomonas aeruginosa* (gram negative), *Staphylococcus* (gram positive)) وقد تبين من النتائج بوجود الحساسية العالية للمركبات المحضرة تجاه نوعي من البكتريا.