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Preparation and Characterization of Derivatives of Pyrimidines in two ways Clascical and Microwave

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Abstract:

This study includes synthesis and characterization of new pyrimidine derivatives (R or Ar-2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one), via of the reaction from cyclohexane-1,3-dione with aldehyde derivatives and guanidine hydrochlorid. Using absolute ethanol as a solvent .This mixture was refluxed for (4 – 6) hrs; While maintaining the pH at 6. The same derivatives of pyrimidine were prepared in microwave way technique. The prepared compounds were characterized by melting point , FT-IR , UV-Vis ^{1}H-NMR and ^{13}C-NMR spectroscopy .

Key Words: Preparation , Characterization , Derivatives , Pyrimidines , Clascical , Microwave

1. Introduction

Pyrimidines were recognized in their antifungal [١,٢] anticancer [٣,٤] and antimicrobial [٥,٧]. Pyrimidines are heterocycle organic aromatic compounds [8]. The basic skeleton of a pyrimidine ring composed of two nitrogen in 1,3-position and carbon atoms . They are also named as diazines 1 and 3.[9]

The significant position of pyrimidine and its derivatives in organic chemistry is primarily related to their bio activity. Above of all, that's, the constitute of nucleic acids which are the base of life, three nitrogenous base (cytosine, thymine and uracil) are pyrimidine derivatives[10]. The biodynamic property of pyrimidine ring structure has urged the medicinal chemists to synthesize such pyrimidine derivatives which can stimulate pharmacophores to utilize it for various pharmacological application. The core structure of pyrimidine helps them by offering certain reaction sites that can be used to reach further with different moieties [11,12].The present research was designed to synthesize derivatives of pyrimidine compounds adopting conventional synthesis reported in literature and the microwave technique that is assisted synthesis[13]. Microwave assisted synthesis is acknowledged a major breakthrough synthetic chemistry in recent years. This technique has overcome the certain backdraws associated with conventional routes i.e. long reaction time, lower yields , purity and slow rate of reaction[14] .

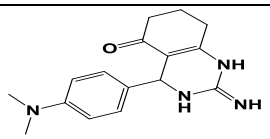
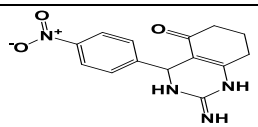
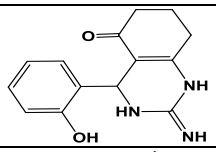
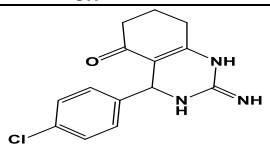
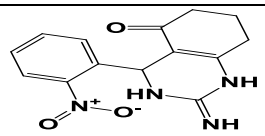
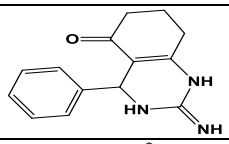
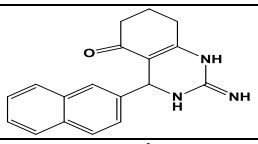
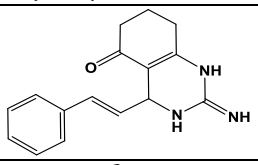
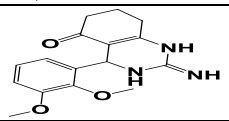
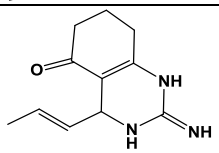
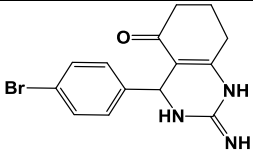
The use of microwave irradiation is the alternative heating technique in synthetic chemistry. Microwave synthesis provides more opportunities to organic chemists to expand their synthetic avenues by applying microwave irradiation to a variety of organic reactions with improved results [15,17].

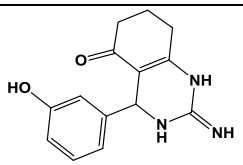
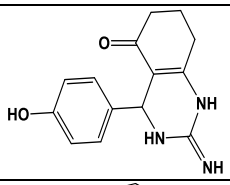
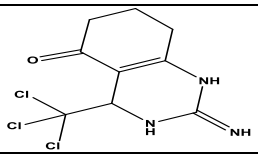
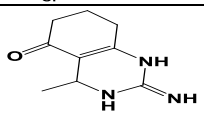
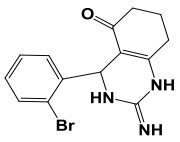
2. Material and Methods:

Preparation of Pyrimidines Derivatives [18]

A series of pyrimidine derivatives were synthesized by the reaction of cyclohexane-1,3-dione (3.36 g , 0.03mol) with aldehyde derivatives (0.025mole) and guanidine hydrochlorid (2.1 g , 0.022 mole) in ethanol absolute (100 ml). This mixture was refluxed for (4-6) hrs. at (90 C^{o}) few drops of hydrochlorid acid to keep pH at (6) the end of the reaction was checked by (T.L.C.). The sludge was filtered recrystallized from ethanol and preserved. All physical properties are listed in table (1).

Table [1]: Physical properties of compounds [C1 – C16] prepared by conventional method.

Comp. No	Structure of the compounds	Color	Yield%	M.P	Time of reaction	aldehydes' Weight (g)or(ml)
C1		orange	96	159-161	4h	3.05 g
C2		yellow	78	148-150	5h	3.78 g
C3		Light Brown	78	220-222	5h	3.05 g
C4		Light yellow	55	242-244	4h	3.5 g
C5		Light Brown	85	152-154	5h	3.78 g
C6		white	90	267-269	5h	2.5 ml
C7		Yellow-greenish	98	194-196	5h	3.6 ml
C8		Light yellow	56	136-138	6h	3.1 ml
C9		Browne	95	oily	6h	3.19 g
C10		Light Brown	85	oily	5h	2.07 ml
C11		dark Brown	80	oily	5h	4.63 g

C12		Light Brown	80	oily	4h	3.05 g
C13		Brown-reddish	60	146-148	6h	3.05 g
C14		Light yellow	48	oily	9h	2.62ml
C15		yellow	95	oily	4h	1.4 ml
C16		yellow	80	187-189	5h	2.92 ml

Microwave Assisted Synthesis of Pyrimidine Derivatives. (R or Ar - 2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one)

The same method used for conventional preparation was used to prepare the compound using the microwave technique except 10 ml of ethanol was added instead of 100 ml.The physical properties and the time of reactions for the compounds were listed in table [2].

Table [2]: Physical properties and time of compounds [C1 – C16]

Comp. No.	Nomenclature	Chemical formula	Time of reaction Min.	M. wt. (g/mol)
C1	4-(4-(dimethylamino)phenyl)-2-imino-2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one	$C_{16}H_{20}N_3O$	0:05	284.36
C2	2-imino-4-(4-nitrophenyl)-2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one	$C_{14}H_{14}N_4O_3$	0:38	286.29
C3	4-(2-hydroxyphenyl)-2-imino-2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one	$C_{14}H_{15}N_3O_2$	0:10	257.29
C4	4-(4-chlorophenyl)-2-imino-2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one	$C_{14}H_{14}ClN_3O$	0:13	275.74
C5	2-imino-4-(2-nitrophenyl)-2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one	$C_{14}H_{14}N_3O$	0:31	286.29
C6	2-imino-4-phenyl-2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one	$C_{14}H_{15}N_3O$	0:51	241.29
C7	2-imino-4-(naphthalen-2-yl)-2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one	$C_{18}H_{17}N_3O$	0:09	291.35
C8	(E)-2-imino-4-styryl-2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one	$C_{16}H_{17}N_3O$	0:13	267.33
C9	4-(2,3-dimethoxyphenyl)-2-imino-	$C_{16}H_{19}N_3O_3$	0:12	301.35

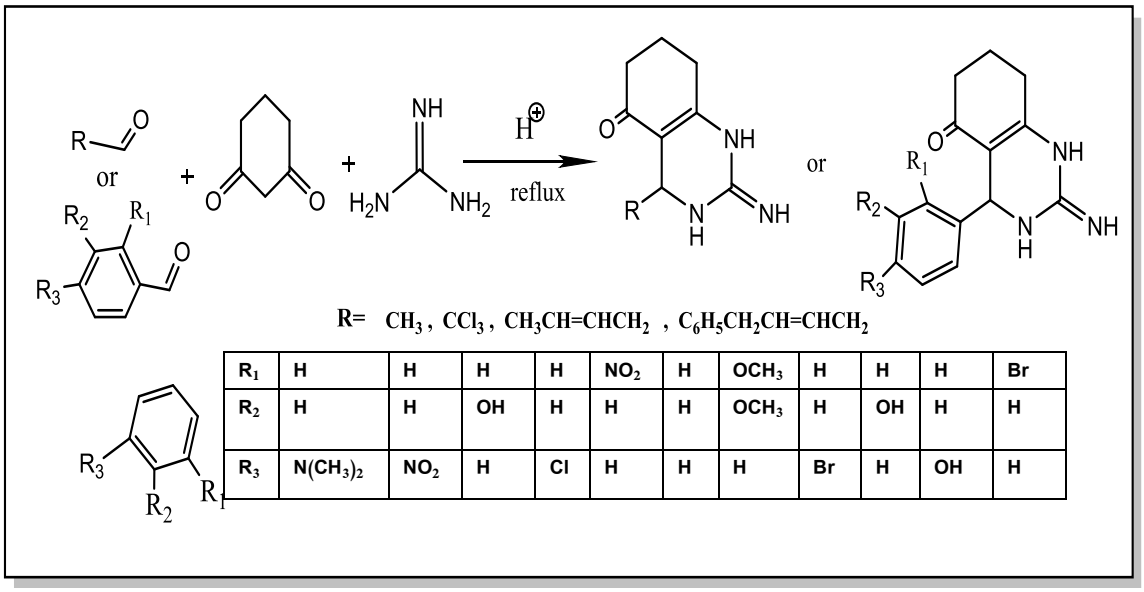
	2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one			
C10	(E)-2-imino-4-(prop-1-en-1-yl)-2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one	$C_{11}H_{15}N_3O$	0:11	203.26
C11	4-(4-bromophenyl)-2-imino-2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one	$C_{14}H_{14}BrN_3O$	0:33	320.19
C12	4-(3-hydroxyphenyl)-2-imino-2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one	$C_{14}H_{15}N_3O_2$	0:11	257.29
C13	4-(4-hydroxyphenyl)-2-imino-2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one	$C_{14}H_{15}N_3O_2$	0:10	257.29
C14	2-imino-4-(trichloromethyl)-2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one	$C_9H_{10}Cl_3N_3O$	2:00	282.55
C15	2-imino-4-methyl-2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one	$C_9H_{13}N_3O$	2:00	179.22
C16	4-(2-bromophenyl)-2-imino-2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one	$C_{14}H_{14}BrN_3O$	0:16	320.19

3. Results and Discussions :

The compounds [C1-C16] were prepared by the reaction of cyclohexane-1,3-dione; different aldehydes and guanidine hydrochlorid in the presence of HCl and absolute ethanol , as show in schem(1).

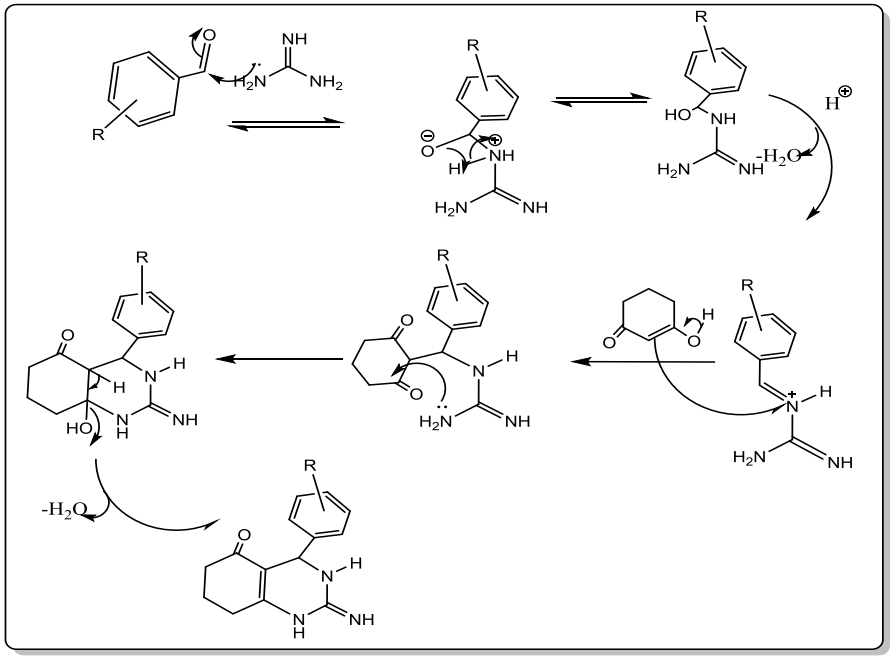
The mechanism involves nucleophilc attack of amino group of guanidine hydrochlorid on carbonyl group of banzaldehyde followed by elimination of water molecule. as shown in the following scheme(2)

The structure of compound was identified by FT-IR , ^{1}H-NMR , ^{13}C-NMR and U.V spectrum .The FT-IR spectrum of compound [C2] Fig(1), table [2] shows appearing of stretching vibration of (NH) group of amine at(3306) cm^{-1} and increasing frequency of (C=O) to(1708) cm^{-1}, also spectrum shows other bands (3034) cm^{-1} for aromatic (C-H) , also spectrum shows anther band at (2954) cm^{-1} for aliphatic (C-H) ; band at (1517,1454,1421) cm^{-1} due to aromatic (C=C); bands at (1620) cm^{-1} for stretching vibration of (NO_2) group , and bands at (1388) cm^{-1} for stretching vibration of (C-N) group. In compound [C14] ,Fig (2) , C-Cl_3 note three bands (798, 754, 700) because found Neighboring group for C-Cl , appearing of stretching vibration of (NH) group of amine at (3303-3400) cm^{-1} because found deferent Neighboring group.



R_1	H	H	H	H	NO_2	H	OCH_3	H	H	H	Br
R_2	H	H	OH	H	H	H	OCH_3	H	OH	H	H
R_3	$N(CH_3)_2$	NO_2	H	Cl	H	H	H	Br	H	OH	H

Scheme (1). Preparation of pyrimidine derivatives.



Scheme (2) mechanism for the preparation of pyrimidine derivatives

Table (3): FT−IR spectral data of compound [C1_C16]

Com. Code	vs N-H Amine	vs C-H Aromatic	vs C-H Aliphatic	vs C=O	vs C=N	vs C=C Arom.	Others
C1	3400	3099	2958	1664	1604	1550,1519,1478	C-N.. 1124,1168
C2	3306	3034	2954	1708	1620	1517,1454,1421	NO_2.. 1388
C3	3380	3100	2951	1720	1610	1579,1560,1485	OH…3414
C4	3306	3053	2954	1660	1616	1489,1450,1417	C-Cl..768
C5	3354	3090	2897	1700	1600	1550,1521,1458	NO_2…1593
C6	3302	3030	2949	1675	1600	1492,1489,1421	C-N..1117
C7	3300	3044	2947	1678	1622	1495,1454,1425	C-N..1130
C8	3330	3028	2945	1675	1600	1577,1492,1452	C-N..1110
C9	3396	3099	2970	1675	1600	1516,1458,1429	C-0 1160,1134
C10	3300	-----	2890	1699	1635	----------	C-N..1120
C11	3340	3010	2974	1668	1600	1550,1539,1456	C-Br..775
C12	3320	3020	2998	1698	1658	1581,1517,1485	C-OH—3420
C13	3307	3010	2964	1700	1660	1589,1516,1450	C-OH..3425
C14	3365	----	2960	1708	1664	--------	C-Cl.. 798,754 ,700
C15	3340	-----	2980	1675	1650	---------	C-N ..1116
C16	3350	3010	2960	1662	1610	1550,1469,1429	C-Br…742

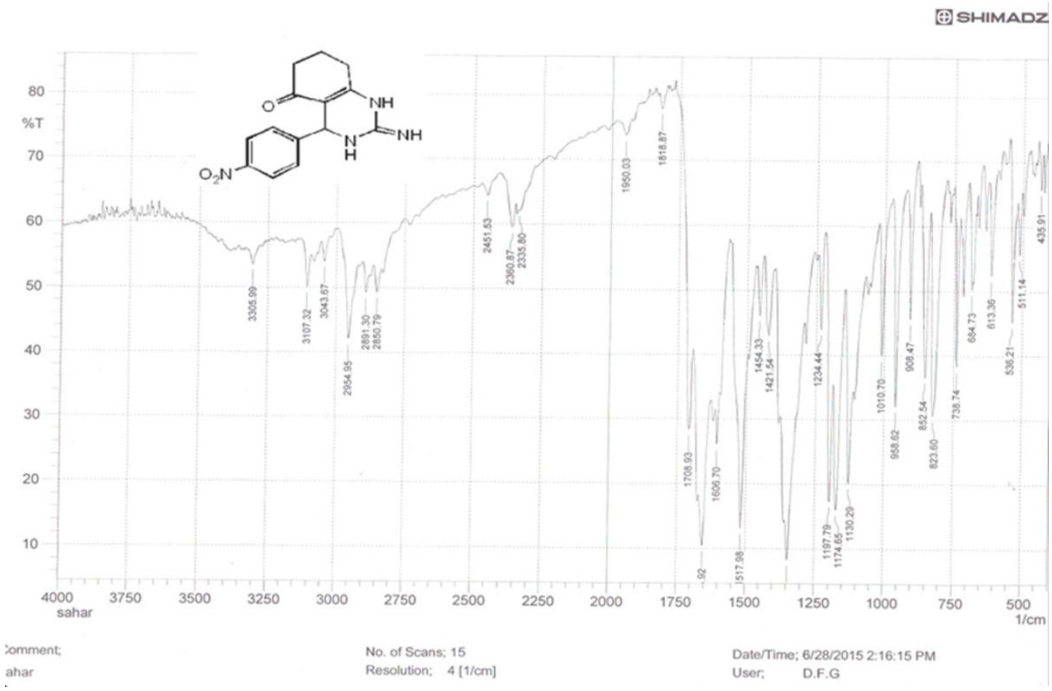


Fig (1): FT-IR spectrum for compound (C2)

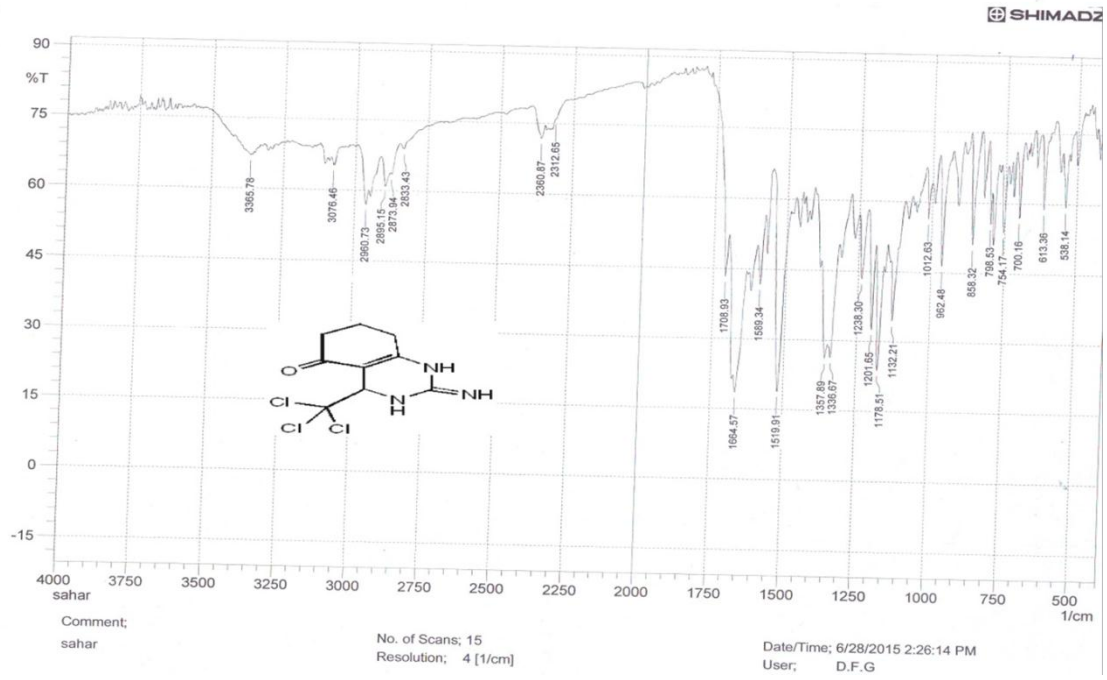


Fig (2): FT-IR spectrum for compound (C14)

By measuring the U.V visible of the compounds prepared by biginelli reaction and microwave method. Shown fig (3), and (4)

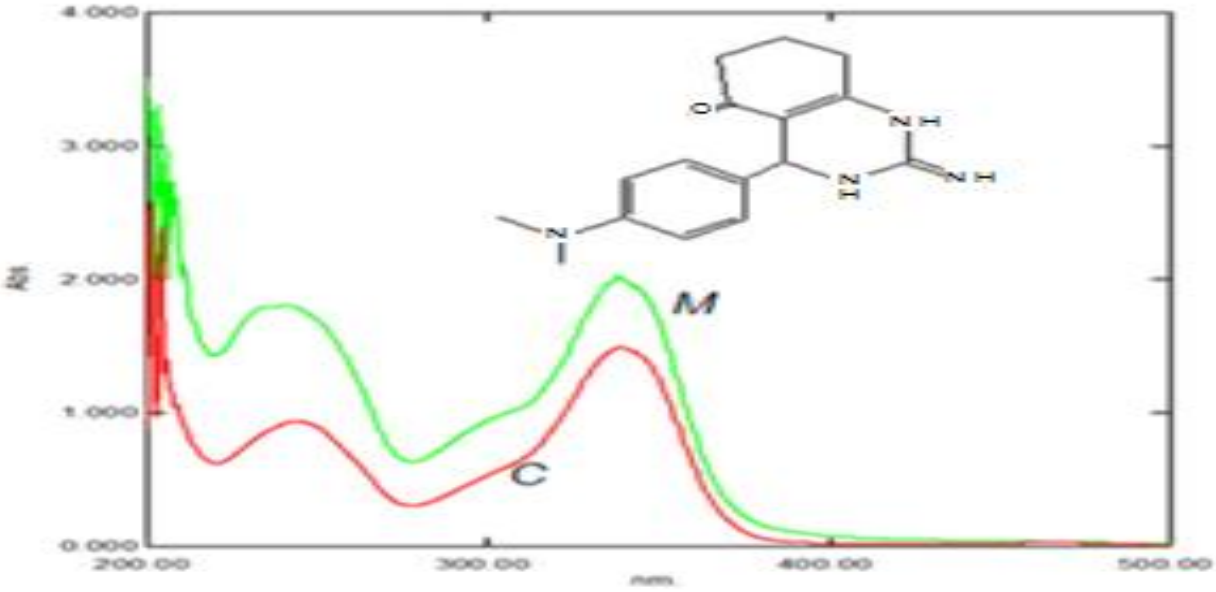


Fig (3) spectrum for compound (C1) M – microwave C – classical

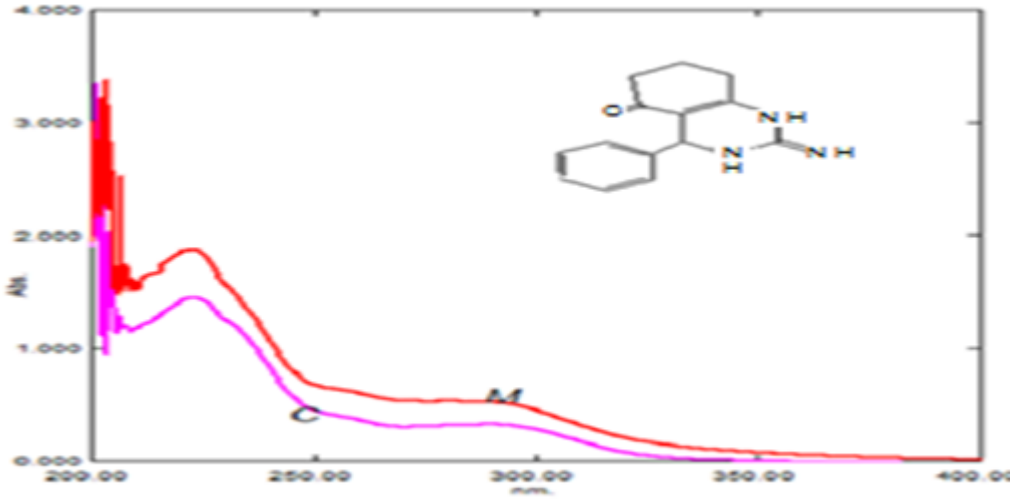


Fig (4) U.V –Visible spectrum for compound (C6) M – microwave C – classical

We noticed that there is matching between these peaks which mean that this method is good, for the preparation but with a little differences

4. Characterization of compounds [C1-C16] by (^{13}C-NMR) for the conventional and microwave methods

The ^{13}C- NMR spectrum for compound (C5) , Fig. (5 a), table [4] shows chemical shift at δ=19.0 ppm assigned to ($CH_2\underline{CH_2}CH_2$). Signal related to ($CH_2CH_2\underline{CH_2}$) was detected at δ=26.47ppm. Signal related to ($CH_2C\underline{H_2}C$=O) was detected at δ=39.01 ppm , Chemical shifts at δ= 41.87 ppm assigned to (HC-N-C) . Signal related (C=NH) was detected at δ=158.13 ppm respectively. Chemical shifts at δ= 127.2- 148.59 ppm attributed to the aromatic ring (C-Ar.). Chemical shift at δ=196.0 ppm assigned to (C=O). The solvent residual signal at δ=40.13 ppm is due to DMSO.(^{13}C-NMR) data for compounds synthesized by both conventional and microwave as shown in fig (5 b),fig (6a) fig (6 b).

Table [4]; ^{13}C-NMR spectral data of compound (C5,C9)

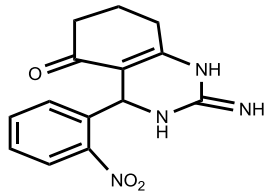
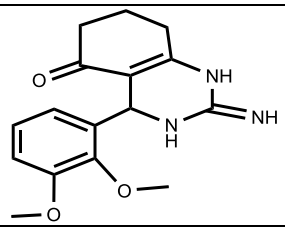
Comp. No.	Compound Structure	^{13}C-NMR Spectral data (δ ppm) M.	^{13}C-NMR Spectral data (δ ppm) C.
C5		δ=20.0 ($CH_2\underline{C}H_2CH_2$) δ=26.49 ($CH_2CH_2\underline{C}H_2$-) , δ=36.34($CH_2\underline{C}H_2C$=O) , δ=41.8 (N-<u>CH</u>-C), δ= 127.29- 149.15 (<u>C</u>-Ar) , δ=149.06 (<u>C</u>=NH) , δ=159.70 (C=<u>C</u>-NH) , δ=196.1 (<u>C</u>=O)	δ=19.0 ($CH_2\underline{C}H_2CH_2$) δ=26.47 ($CH_2CH_2\underline{C}H_2$) , δ=39.01 ($CH_2\underline{C}H_2C$=O) , δ=41.87 (<u>H C</u>-N-C) , δ= 127.2- 148.5 (<u>C</u>-Ar) , δ=158.13 (<u>C</u>=NH), δ=159.19 (C=<u>C</u>-NH), δ=196.0 (<u>C</u>=O)
C9		δ=20.0 ($CH_2\underline{C}H_2CH_2$), δ=28.00 ($CH_2CH_2\underline{C}H_2$), δ=36.41 ($CH_2\underline{C}H_2C$=O) , δ=41.76 (<u>HC</u>-N-C) , δ=55.35-55.39(.$\underline{H_3C}$-O), δ= 119.95-148.38 (<u>C</u>-Ar), δ=157.85 (<u>C</u>=NH)	δ=20.0 ($CH_2\underline{C}H_2CH_2$), δ=30.00 ($CH_2CH_2\underline{C}H_2$), δ=36.41 ($CH_2\underline{C}H_2C$=O) , δ=41.68 (<u>HC</u>-N-C) , δ=55.33-55.58(.$\underline{H_3C}$-O), δ= 113.95-148.87 (<u>C</u>-Ar), δ=157.86 (<u>C</u>=NH)

fig.(5a) ; ^{13}C-NMR spectrum for compound (C5) conventional way

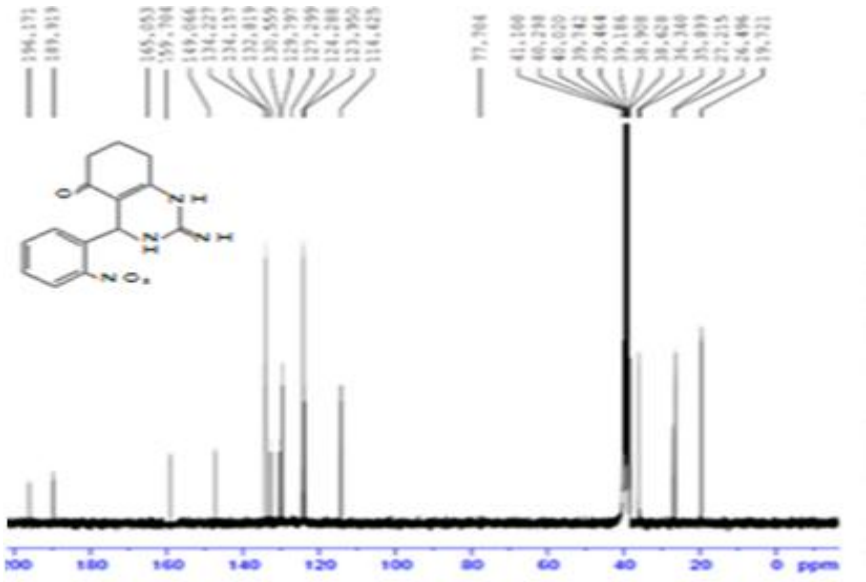


fig.(5b) ; ^{13}C-NMR spectrum for (C5) microwave way compound

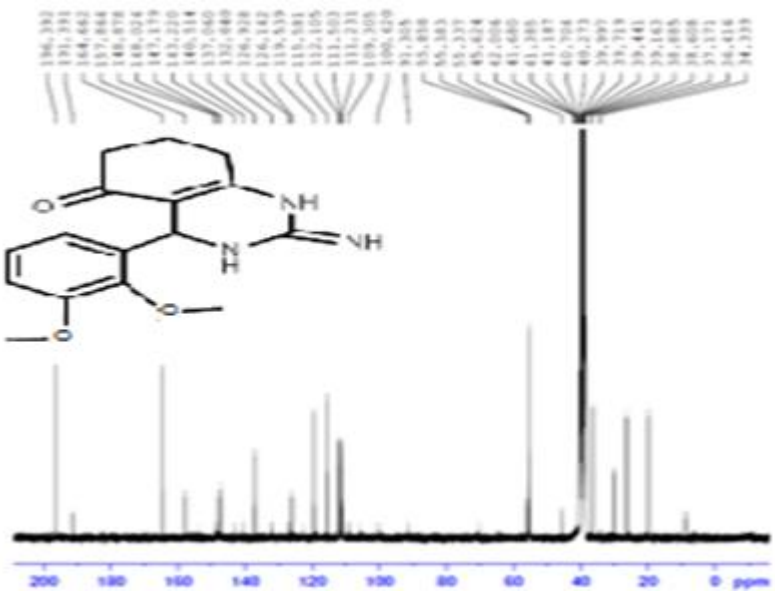


fig.(6a) ; ^{13}C−NMR spectrum for compound (C5) conventional way

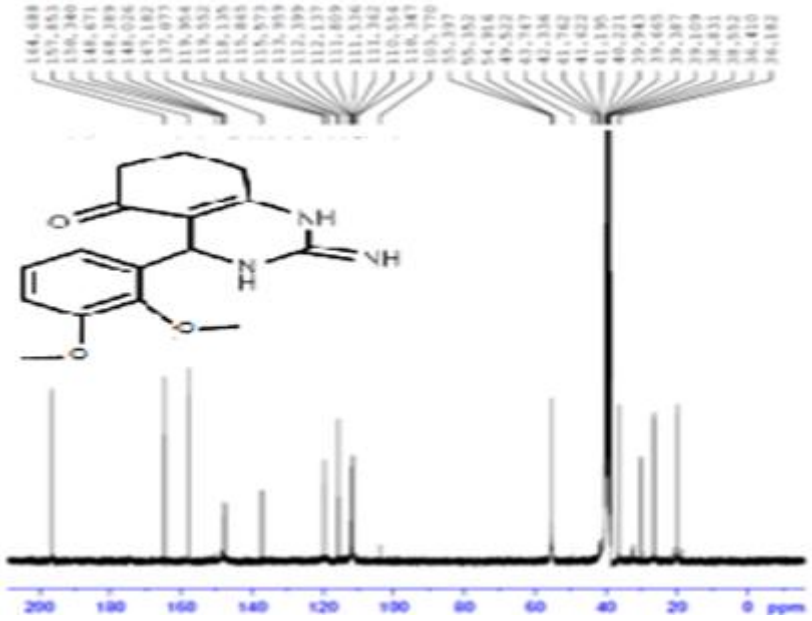
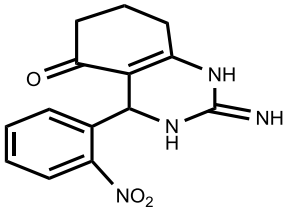
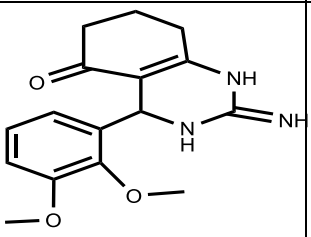


fig.(6b) ; ^{13}C−NMR spectrum for compound (C5) microwave way

5. Characterization of compounds [C1-C16] by (^{1}H-NMR) for the conventional and microwave methods

The ^{1}H NMR spectrum for compound (C5) , Fig. (7a), shows chemical shift at δ=1.4 ppm which assigned to (q,2H,$CH_2\underline{CH_2}CH_2$), signal at δ=1.9 ppm equivalent to protons have been assigned to (t,2H,$\underline{CH_2}CH_2CH_2$) proton. Chemical shift at δ=3.0 ppm which assigned to(s,2H,$\underline{CH_2}$CO) , signal at δ=3.17ppm equivalent to protons have been assigned to (t,1H,NH$\underline{CH}CH_2$) proton .Chemical shift at δ=7.9-8.16ppm assigned to aromatic para- ring substitution protons (m,4H,$\underline{H}$-Ar) protons . Chemical shift at δ=7.78ppm equivalent to One protons has been assigned to (s,1H,C=$\underline{NH}$) protons . This peak is appeared as expected downfield (from TMS chemical shift) .The solvent residual signal at δ=2.5 ppm is due to DMSO ,as shown table [5]. ^{1}H-NMR data for compounds synthesized by both conventional and microwave, as shown in fig (7b) , fig (8a),fig (8b).

Table [5]: ^{1}H-NMR spectral data of compound (C5, C9)

Comp. No.	Compound Structure	^{1}H-NMR Spectral data (δ ppm) M.	^{1}H-NMR Spectral data (δ ppm) C.
C5		δ=1.4(q,2H,$CH_2\underline{CH_2}CH_2$), δ=1.9(t,2H,$\underline{CH_2}CH_2CH_2$), δ=3.0(s,2H,$\underline{CH_2}$CO), δ=3.17(s,2H,$\underline{HC}$-N-C), δ=7.9-8.16(m,4H,Ar), δ=7.78(s,1H,C=$\underline{NH}$), δ=10.5 (s,1H,C=C-$\underline{NH}$)	δ=1.4(q,2H,$CH_2\underline{CH_2}CH_2$), δ=2.1(t,2H,$\underline{CH_2}CH_2CH_2$-), δ=3.1(s,2H,$\underline{CH_2}$CO), δ=3.9(s,2H,$\underline{HC}$-N-C), δ=7.51-7.93(m,4H,Ar), δ=7.85(s,1H,C=$\underline{NH}$), δ=10.5 (s,1H,C=C-$\underline{NH}$)
C9		δ=1.3(q,2H,$CH_2\underline{CH_2}CH_2$), δ=1.8(t,2H,$\underline{CH_2}CH_2CH_2$), δ=3.8(s,3H,H_3C-O), δ=4.58(s,1H,H_3C-$\underline{NH}$-C), δ=6.82-6.93.(m,3H,$\underline{H}$-Ar), δ=7.2(s,1H,C=N$\underline{H}$), δ=10.1(s,1H,C-N$\underline{H}$-C)	δ=1.2(q,2H,$CH_2\underline{CH_2}CH_2$), δ=2.0(t,2H,$\underline{CH_2}CH_2CH_2$), δ=3.82(s,3H,H_3C-O), δ=3.86(s,3H,$\underline{H_3C}$-N-C), δ=4.53(s,1H,H_3C-$\underline{NH}$-C), δ=6.91-6.96(m,3H,$\underline{H}$-Ar), δ=7.57-8.64(s,1H,C=N$\underline{H}$), δ=9.87 (C-N$\underline{H}$-C)

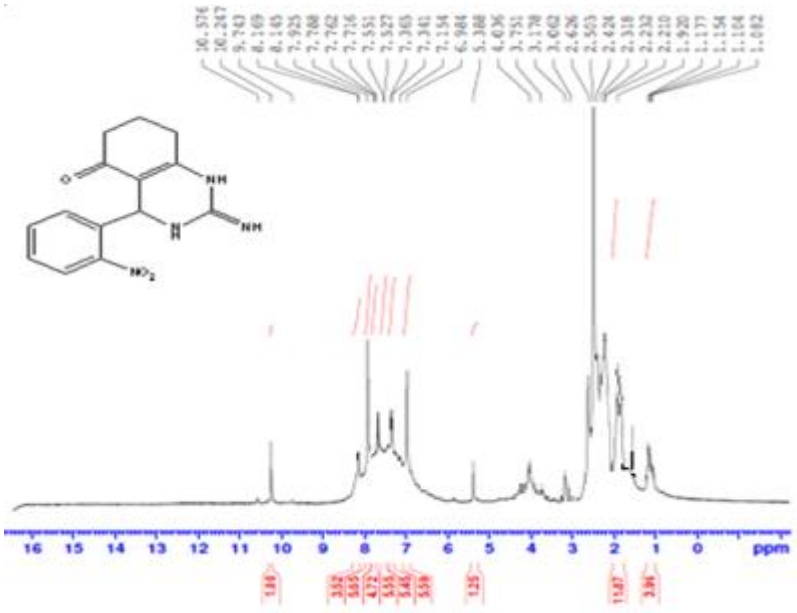


fig.(7a) ; ^{1}H-NMR spectrum for compound (C5) conventional way

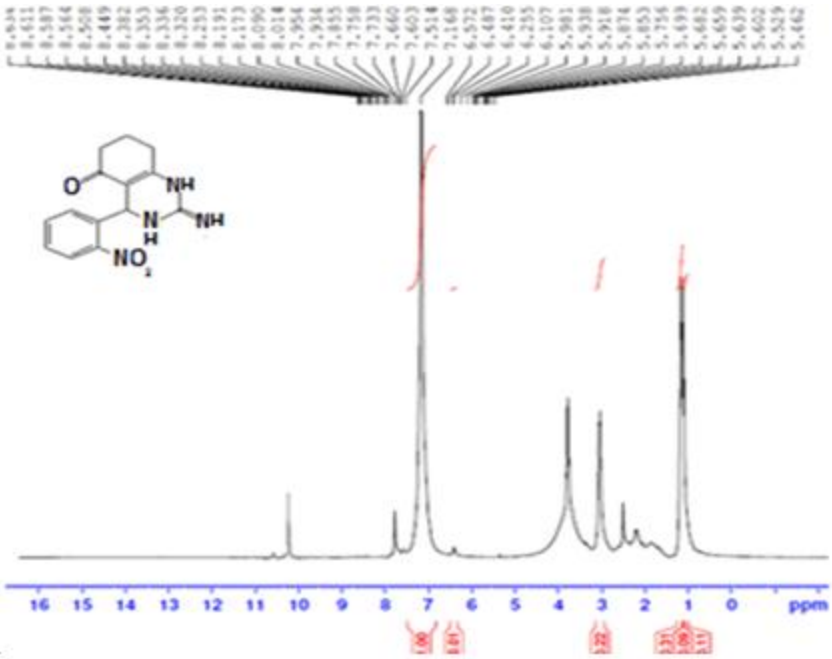


fig.(7b) ; ^{1}H-NMR spectrum for compound (C5) microwave way

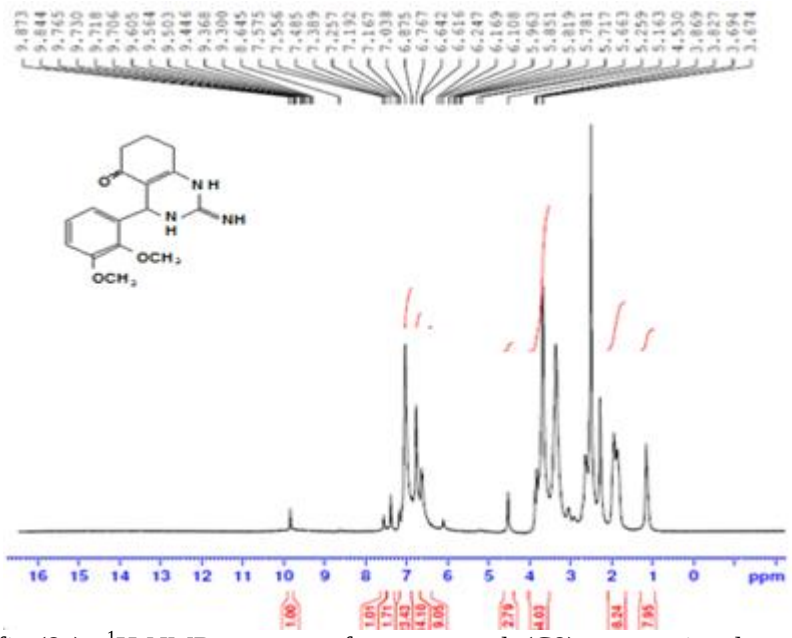


fig.(8a) ; ^{1}H-NMR spectrum for compound (C9) conventional way

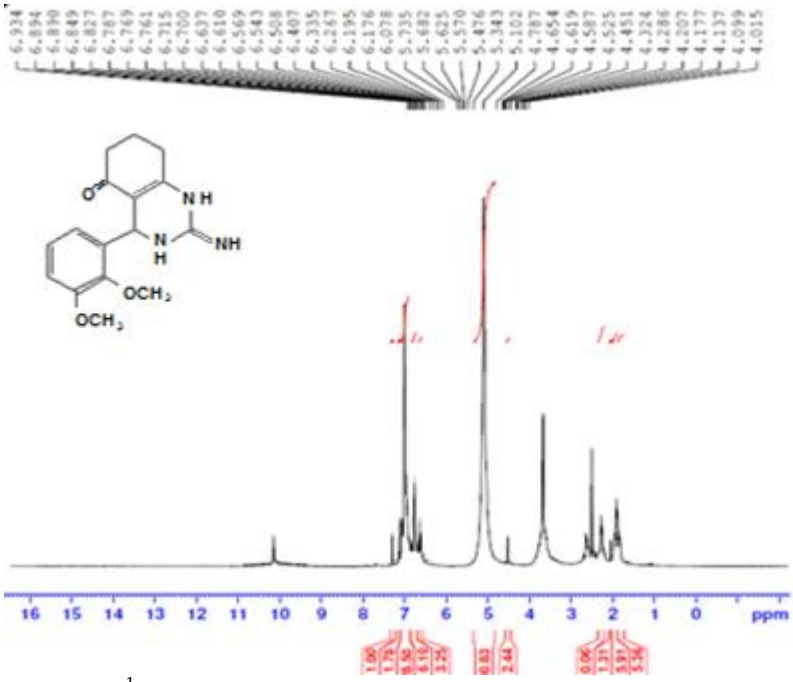


fig.(8b) ; ^{1}H-NMR spectrum for compound (C9) microwave way

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تحضير وتشخيص مشتقات البريميدنات بطريقتي الكلاسيكية والمايكروويف

وليد فرج حمادي سميعة جمعة خماس بشرى تركي مهدي

الخلاصة

اشتمل هذا البحث تحضير وتشخيص بعض مشتقات البريميدين (الكيل أو اريل –8,7,6,4,3,2–سداسي هايدور كوينوزولين 5–(1H) –اون) عبر تفاعل هكسان الحلقي– 3,1,– داي اون مع الديهايدات معوضة و الكواندين هيدروكلورايد. أستخدام الايثانول المطلق كمذيب ويستمر تصعيد التفاعل من (4–6) ساعة. مع المحافظة على الدالة الحامضية الهيدروجيني عند pH 6 . حضرت البريمدينات السابقة ايضاً بطريقة المايكرويف وتم تشخيص المركبات المحضرة بقياس درجة انصهار والاشعة تحت الحمراء والاشعة فوق البنفسجية وأطياف الرنين النووي المغناطيسي لنوى الهيدروجين (^{1}H–NMR) والرنين النووي المغناطيسي لذرة الكاربون 13 (^{13}C–NMR) .