

Preparation and Characterization of Derivatives of Pyrimidines in two ways Classical 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 guanidinehydrochlorid. 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 ¹H-NMR and ¹³C-NMR spectroscopy.

1. Introduction

Pyrimidines were recognized in their antifungal (1,2) anticancer (3,4) and antimicrobial (5,7). Pyrimidines are heterocyclic 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 in synthetic chemistry in recent years. This technique has overcome the certain drawbacks 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).

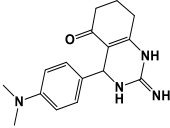
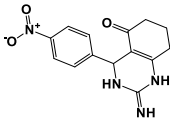
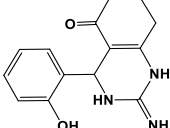
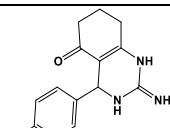
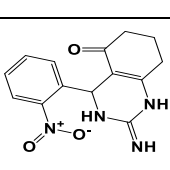
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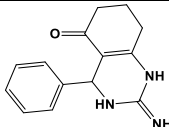
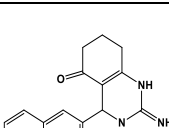
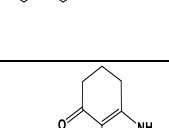
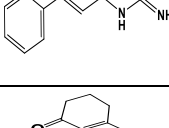
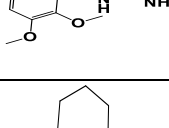
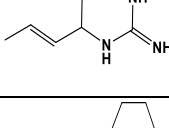
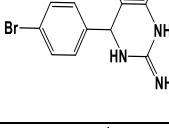
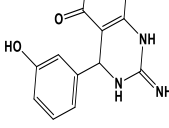
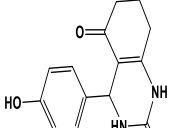
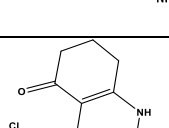
2. Material and Methods:

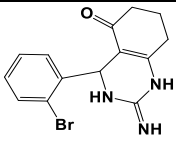
Preparation of Pyrimidines Derivatives⁽¹⁸⁾

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 guanidinehydrochlorid (2.1 g , 0.022 mole) in ethanol absolute (100 ml). This mixture was refluxed for (4-6) hrs. at (90 C°) 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
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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 ₃ O	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 ₃ O 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 ₃ O 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 ₃ O	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 ₃ O	0:31	286.29
C6	2-imino-4-phenyl-2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one	C ₁₄ H ₁₅ N ₃ O	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 ₃ O	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 ₃ O	0:13	267.33
C9	4-(2,3-dimethoxyphenyl)-2-imino-2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one	C ₁₆ H ₁₉ N ₃ O ₃	0:12	301.35
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 ₃ O	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 ₃ O	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 ₃ O 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 ₃ O ₂	0:10	257.29
C14	2-imino-4-(trichloromethyl)-2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one	C ₉ H ₁₀ Cl ₃ N ₃ O	2:00	282.55
C15	2-imino-4-methyl-2,3,4,6,7,8-hexahydroquinazolin-5(1H)-one	C ₉ H ₁₃ N ₃ O	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 ₃ O	0:16	320.19

3. Results and Discussions :

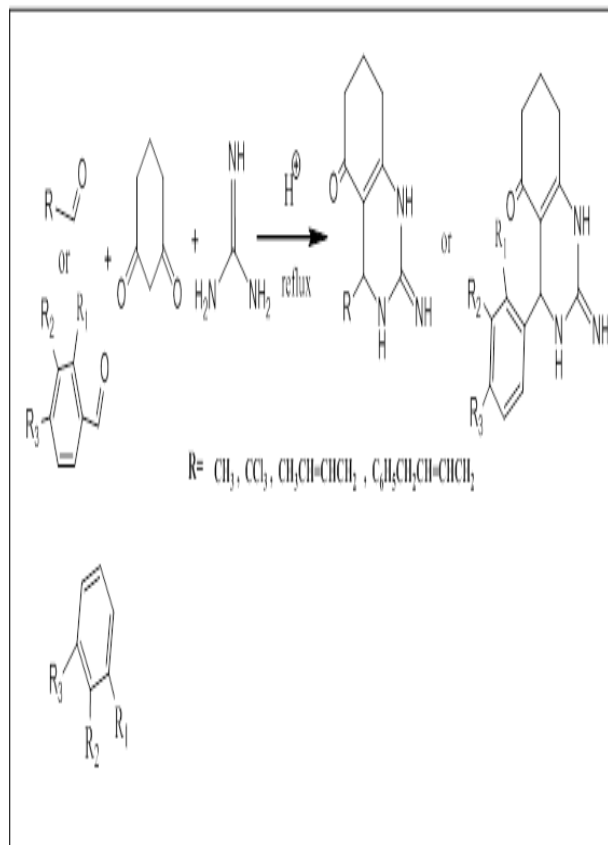
The compounds [C1-C16] were prepared by the reaction of cyclohexane-1,3-dione; different aldehydes

and guanidinehydrochlorid in the presence of HCl and absolute ethanol , as show in schem(1).

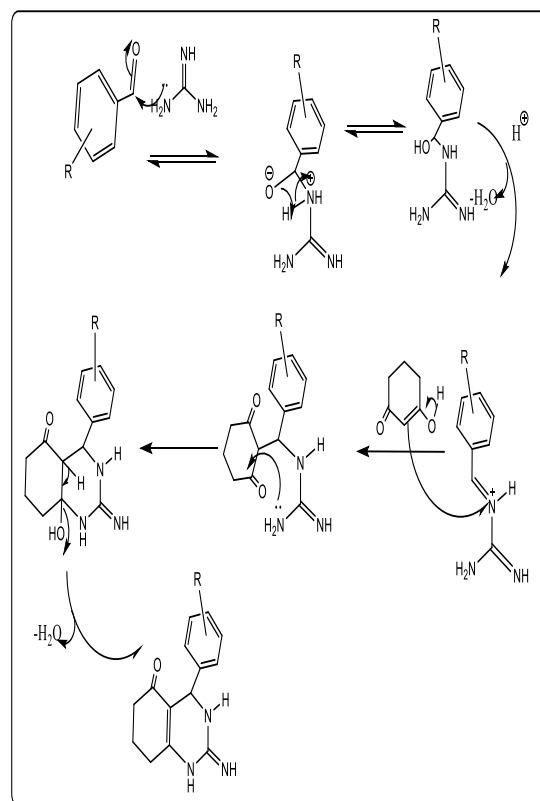
The mechanism involves nucleophilic attack of amino group of guanidine hydrochlorid on carbonyl group of banzaldehyde followed by elimination of water molecule. as shown in the following scheme(2)

The structure of compound was identified by FT-IR , ¹H-NMR , ¹³C-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 (NO₂) group , and bands at (1388) cm⁻¹ for stretching vibration of (C-N) group. In compound [C14] ,Fig (2) , C-Cl₃ 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	R ₂	R ₃
H	H	N(CH ₃) ₂
H	H	NO ₂
H	OH	H
H	H	Cl
NO ₂	H	H
H	H	H
OCH ₃	OCH ₃	H
H	H	Br
H	OH	H
H	H	OH
Br	H	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]

C7	C6	C5	C4	C3	C2	C1	Com. Code
3300	3302	3354	3306	3380	3306	3400	vs N-H Amine
3044	3030	3090	3053	3100	3034	3099	vs C-H Aromatic
2947	2949	2897	2954	2951	2954	2958	vs C-H Aliphatic
1678	1675	1700	1660	1720	1708	1664	vs C=O
1622	1600	1600	1616	1610	1620	1604	vs C=N
1495,1454,1425	1492,1489,1421	1550,1521,1458	1489,1450,1417	1579,1560,1485	1517,1454,1421	1550,1519,1478	vs C=C Arom.
C-N..1130	C-N..1117	NO ₂ ...1593	C-Cl..768	OH...3414	NO... 1388	C-N.. 1124,1168	Others

C16	C15	C14	C13	C12	C11	C10	C9	C8
3350	3340	3365	3307	3320	3340	3300	3396	3330
3010	-----	----	3010	3020	3010	-----	3099	3028
2960	2980	2960	2964	2998	2974	2890	2970	2945
1662	1675	1708	1700	1698	1668	1699	1675	1675
1610	1650	1664	1660	1658	1600	1635	1600	1600
1550,1469,1429	-----	-----	1589,1516,1450	1581,1517,1485	1550,1539,1456	-----	1516,1458,1429	1577,1492,1452
C-Br...742	C-N ..1116	C-Cl.. 798,754, 700	C-OH...3425	C-OH—3420	C-Br..775	C-N..1120	C-0 1160,1134	C-N..1110

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

By measuring the U.V visible of the compounds prepared by biginelli reaction and microwave method.

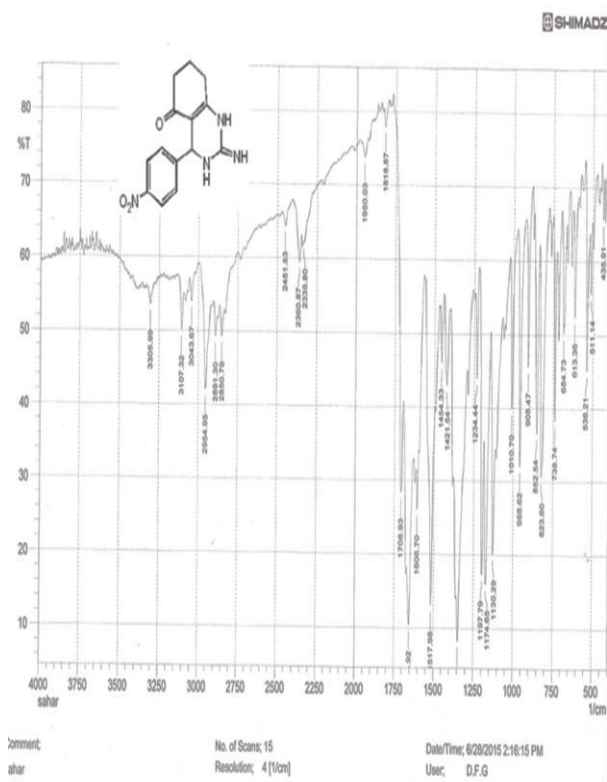
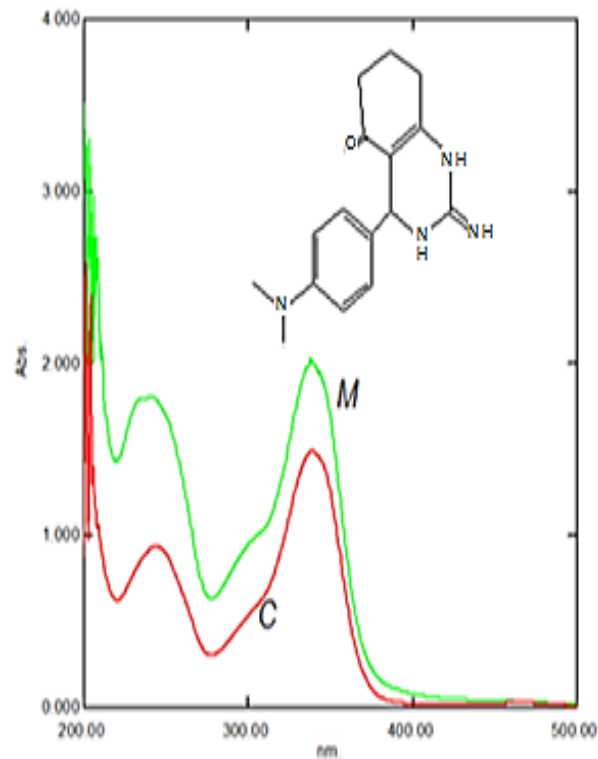
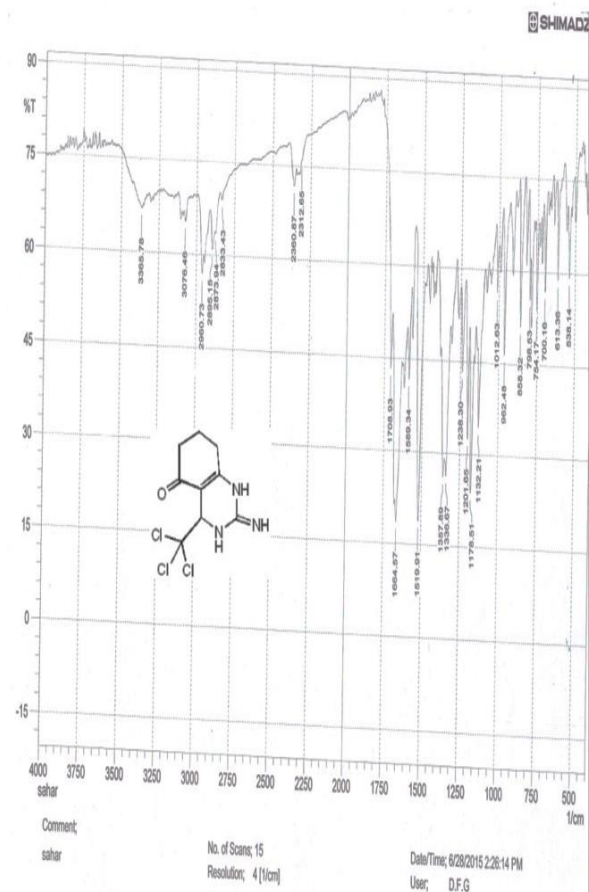


Fig (1): FT-IR spectrum for compound (C2)



Shown fig (3), and (4).

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

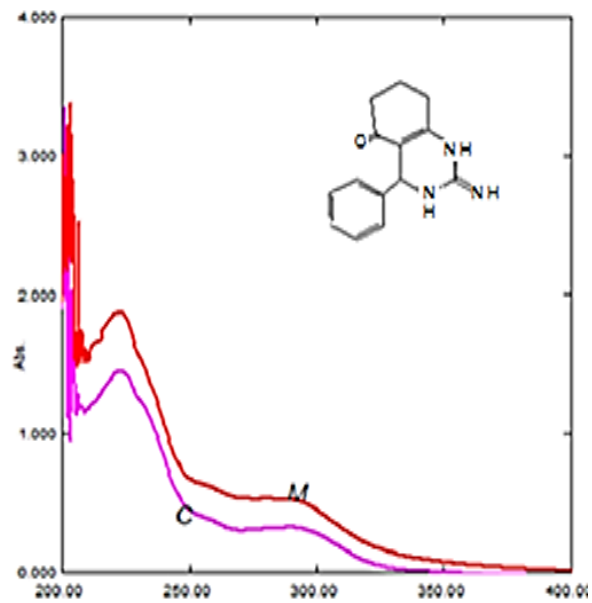


Fig (4) U.V –Visible spectrum for compound (C6) M – microwave C – classical

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 (¹³C-NMR) for the conventional and microwave methods

The ¹³C- NMR spectrum for compound (C5) , Fig. (5a), table [4] shows chemical shift at δ=19.0 ppm assigned to (CH₂CH₂CH₂). Signal related to (CH₂CH₂CH₂) was detected at δ=26.47ppm. Signal related to (CH₂CH₂C=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.(¹³C-NMR) data for compounds synthesized by both conventional and microwave as shown in fig (5 b),fig (6a)fig (6 b).

Table [4]; ¹³C-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.
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C5		$\delta=20.0$ (CH ₂ CH ₂ CH ₂) $\delta=26.49$ (CH ₂ CH ₂ CH ₂), $\delta=36.34$ (CH ₂ CH ₂ C=O) , $\delta=41.8$ (N-CH-C), $\delta= 127.29$ - 149.15 (C-Ar) , $\delta=149.06$ (C=NH) , $\delta=159.70$ (C=C-NH) , $\delta=196.1$ (C=O)	$\delta=19.0$ (CH ₂ CH ₂ CH ₂) $\delta=26.47$ (CH ₂ CH ₂ CH ₂) , $\delta=39.01$ (CH ₂ CH ₂ C=O) , $\delta=41.87$ (HC-N-C) , $\delta= 127.2$ - 148.5 (C-Ar) , $\delta=158.13$ (C=NH) , $\delta=159.19$ (C=C-NH), $\delta=196.0$ (C=O)
C9		$\delta=20.0$ (CH ₂ CH ₂ CH ₂) , $\delta=28.00$ (CH ₂ CH ₂ CH ₂) , $\delta=36.41$ (CH ₂ CH ₂ C=O) , $\delta=41.76$ (HC-N-C) , $\delta=55.35$ - 55.39 (H ₃ C-O) , $\delta=$ 119.95 - 148.38 (C-Ar) , $\delta=157.85$ (C=NH)	$\delta=20.0$ (CH ₂ CH ₂ CH ₂) , $\delta=30.00$ (CH ₂ CH ₂ CH ₂) , $\delta=36.41$ (CH ₂ CH ₂ C=O) , $\delta=41.68$ (HC-N-C) , $\delta=55.33$ - 55.58 (H ₃ C-O) , $\delta=$ 113.95 - 148.87 (C-Ar) , $\delta=157.86$ (C=NH)

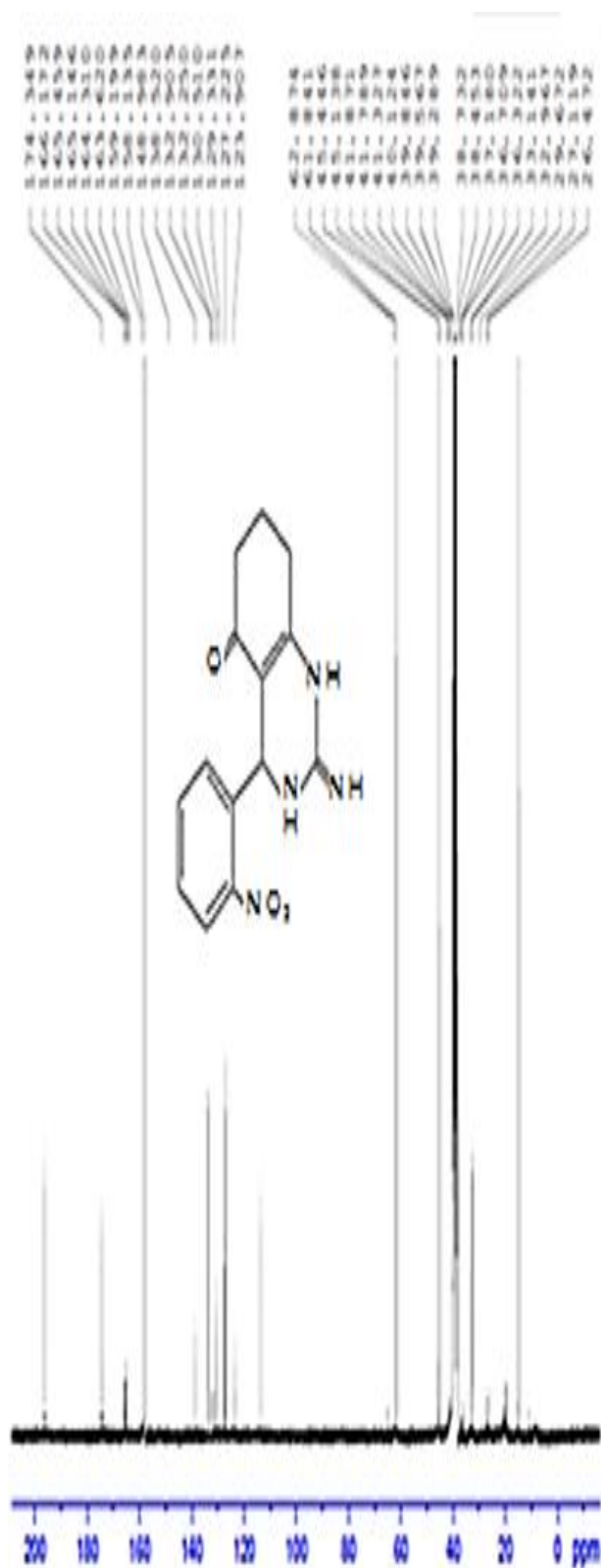


fig.(5a) ; ¹³C-NMR spectrum for compound(C5)
conventional way

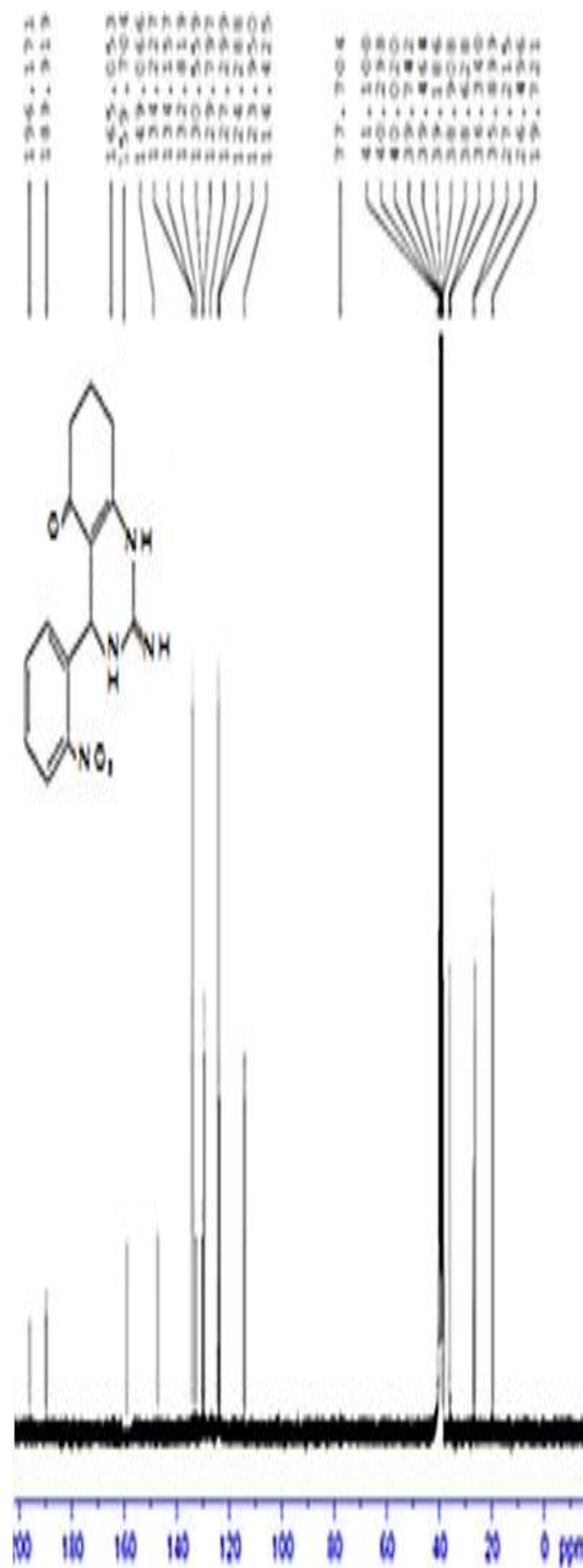


fig.(5b) ; ¹³C-NMR spectrum for (C5) microwaveway
compound

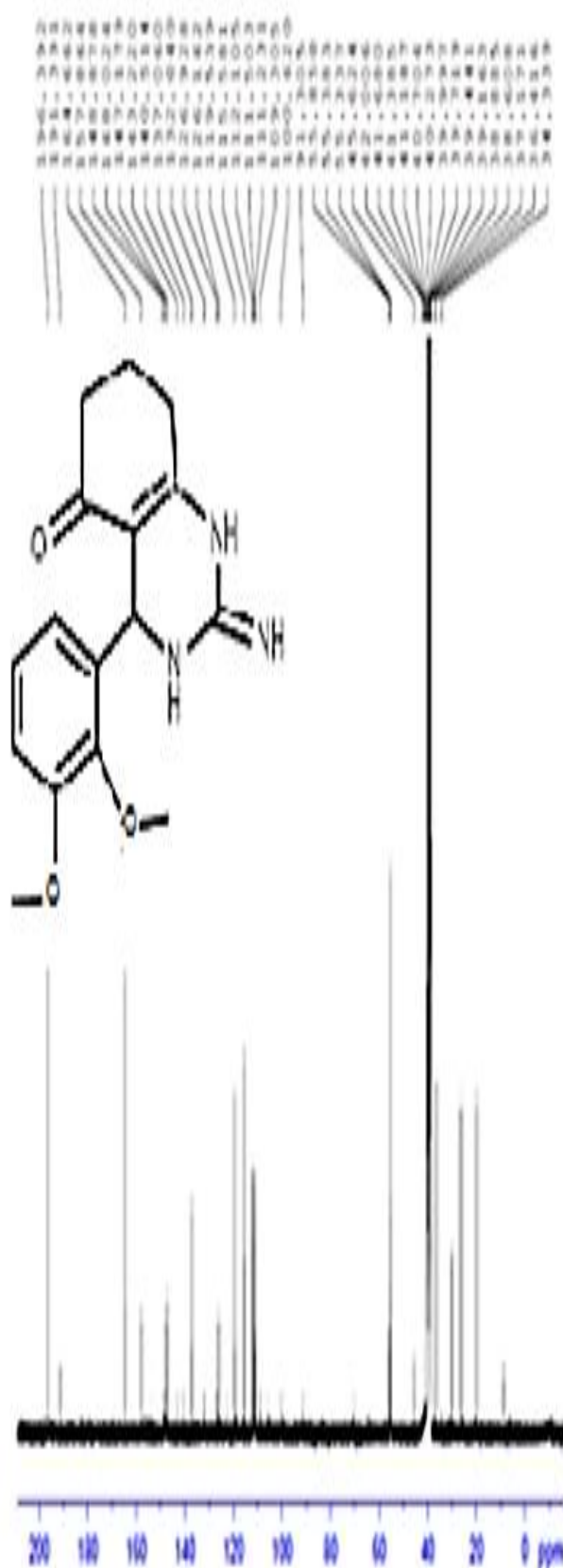


fig.(6a) ; ¹³C-NMR spectrum for compound (C5)
conventional way

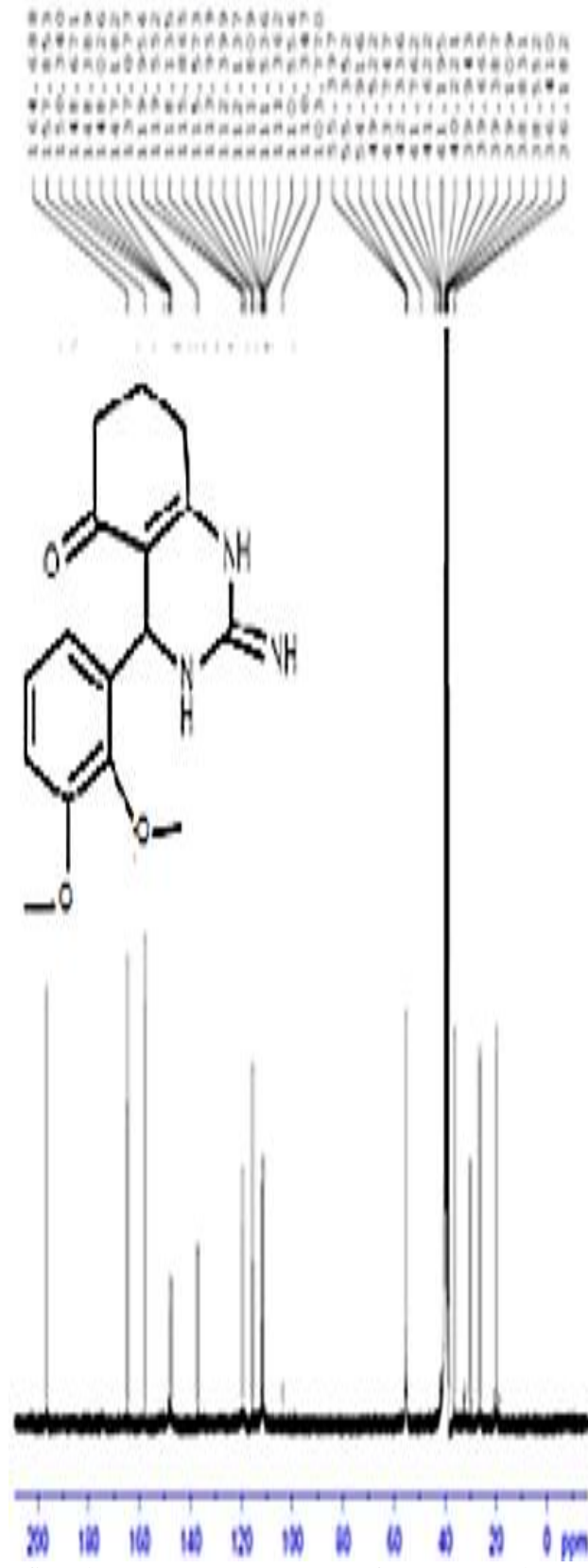


fig.(6b) ; ¹³C-NMR spectrum for compound (C5)
microwave way

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

The ¹H NMR spectrum for compound (C5) , Fig. (7a), shows chemical shift at $\delta=1.4$ ppm which assigned to (q,2H,CH₂CH₂CH₂), signal at $\delta=1.9$ ppm equivalent to protons have been assigned to (t,2H,CH₂CH₂CH₂) proton. Chemical shift at $\delta=3.0$ ppm which assigned to (s,2H,CH₂CO) , signal at $\delta=3.17$ ppm equivalent to protons have been assigned to (t,1H,NHCHCH₂) proton .Chemical shift at $\delta=7.9-8.16$ ppm assigned to aromatic para- ring substitution protons (m,4H,H-Ar) protons . Chemical shift at $\delta=7.78$ ppm equivalent to One protons has been assigned to (s,1H,C=NH) protons . This peak is appeared as expected downfield (from TMS chemical shift) .The solvent residual signal at $\delta=2.5$ ppm is due to DMSO ,as shown table [5].¹H-NMR data for compounds synthesized by both conventional and microwave, as shown in fig (7b) , fig (8a),fig (8b).

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

Comp. No.	Compound Structure	¹ H-NMR Spectral data (° ppm) M.	¹ H-NMR Spectral data (° ppm) C.
C5		$\delta=1.4$ (q,2H,CH ₂ CH ₂ CH ₂), $\delta=1.9$ (t,2H,CH ₂ CH ₂ CH ₂), $\delta=3.0$ (s,2H,CH ₂ CO), $\delta=3.17$ (s,2H,HC-N-C), $\delta=7.9-8.16$ (m,4H,Ar), $\delta=7.78$ (s,1H,C=NH), $\delta=10.5$ (s,1H,C=C-NH)	$\delta=1.4$ (q,2H,CH ₂ CH ₂ CH ₂), $\delta=2.1$ (t,2H,CH ₂ CH ₂ CH ₂), $\delta=3.1$ (s,2H,CH ₂ CO), $\delta=3.9$ (s,2H,HC-N-C), $\delta=7.51-7.93$ (m,4H,Ar), $\delta=7.85$ (s,1H,C=NH), $\delta=10.5$ (s,1H,C=C-NH)

C9		$\delta=1.3$ (q,2H,CH ₂ CH ₂ CH ₂), $\delta=1.8$ (t,2H,CH ₂ CH ₂ CH ₂), $\delta=3.8$ (s,3H,H ₃ C-O), $\delta=4.58$ (s,1H,H ₃ C-NH-C), $\delta=6.82-6.93$.(m,3H,H-Ar), $\delta=7.2$ (s,1H,C=NH), $\delta=10.1$ (s,1H,C-NH-C)	$\delta=1.2$ (q,2H,CH ₂ CH ₂ CH ₂), $\delta=2.0$ (t,2H,CH ₂ CH ₂ CH ₂), $\delta=3.82$ (s,3H,H ₃ C-O), $\delta=3.86$ (s,3H,H ₃ C-N-C), $\delta=4.53$ (s,1H,H ₃ C-NH-C), $\delta=6.91-6.96$ (m,3H,H-Ar), $\delta=7.57-8.64$ (s,1H,C=NH), $\delta=9.87$ (C-NH-C)
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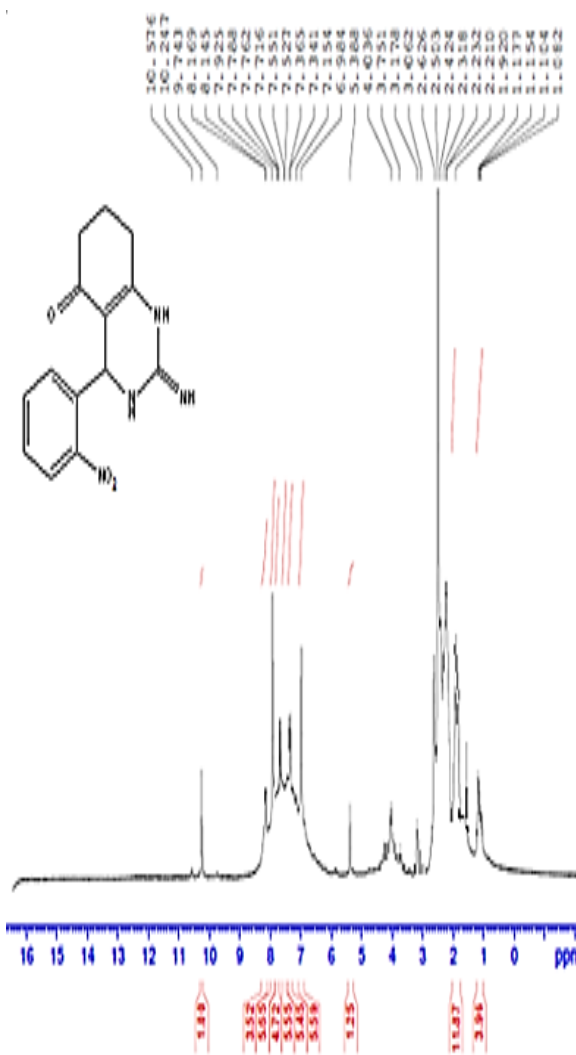


fig.(7a) ; ¹H-NMR spectrum for compound (C5) conventional way

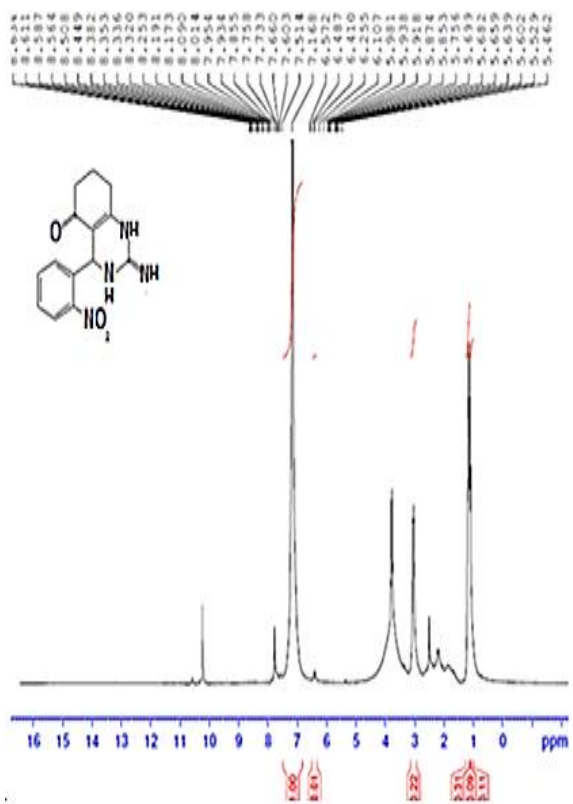


fig.(7b) ; ¹H-NMR spectrum for compound (C5)
microwave way

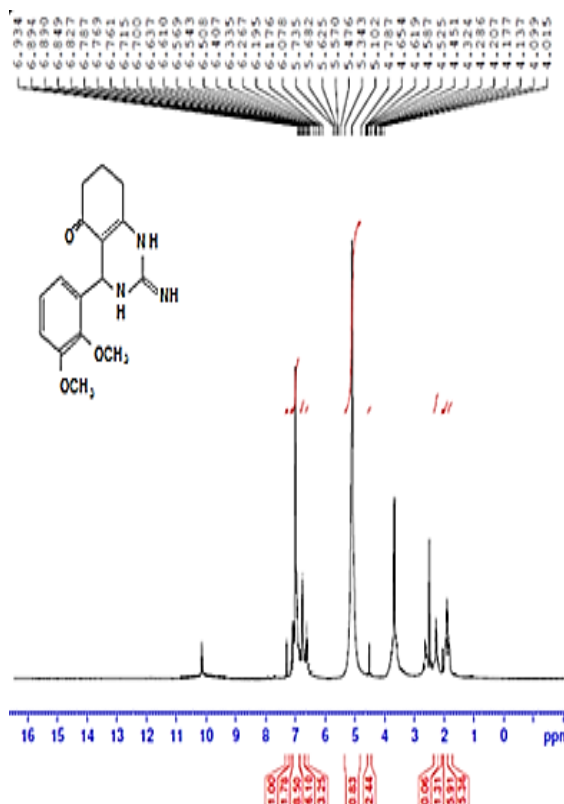


fig.(8b) ; ¹H-NMR spectrum for compound (C9)
microwave way

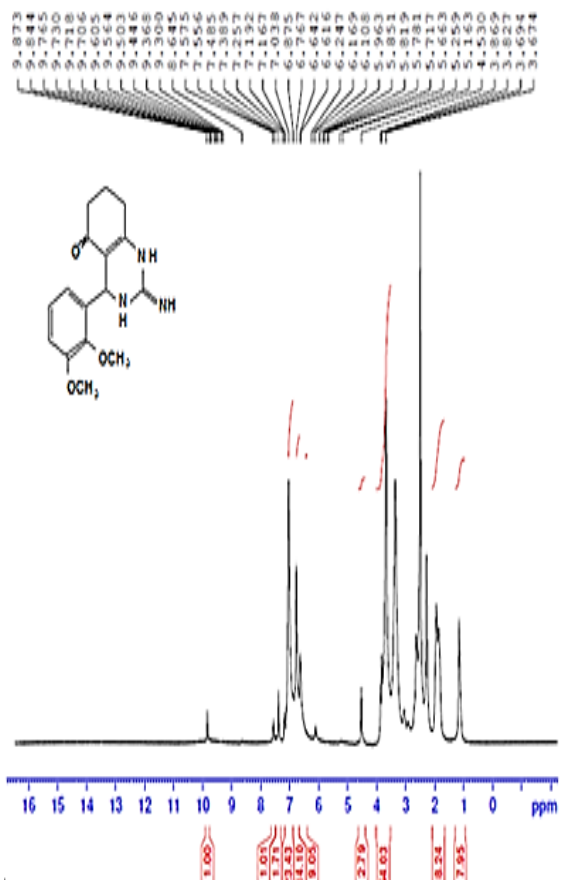


fig.(8a) ; ¹H-NMR spectrum for compound (C9)
conventional way

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تحضير وتشخيص مشتقات البريميدينات بطريقتي الكلاسيكية والمايكروويف

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الخلاصة

اشتمل هذا البحث تحضير وتشخيص بعض مشتقات البريميدين (الكيل أو اريل -2,3,4,6,7,8-سداسي هايدوركوينوزولين-5 (IH)-اون) عبر تفاعل هكسان الحلقي -1,3, -داي اون مع الديهايدانتمعوضة و الكواندينهيدروكلورايد. استخدام الايثانول المطلق كمذيب ويستمر تصعيد التفاعل من (4-6 ساعة) مع المحافظة على الدالة الحامضية الهيدروجيني عند pH 6 حضرت البريميدينات السابقة ايضاً بطريقة المايكروويف وتم تشخيص المركبات المحضرة بقياس درجة انصهار والاشعة تحت الحمراء والاشعة فوق البنفسجية وأطياف الرنين النووي المغناطيسي لنوى الهيدروجين ($^1\text{H-NMR}$) والرنين النووي المغناطيسي لذرة الكربون 13 ($^{13}\text{C-NMR}$).