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A **Synthesis and Characterization of Some Compounds containing Pyrazole Moiety**

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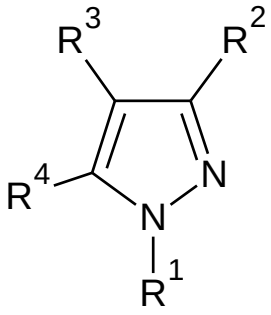
ABSTRACT

This study includes the preparation of some heterocyclic compounds compact of the Pyrazole, work is done in three steps. First step, treatment of acetophenone derivatives with DMF-DMA for the preparation of the first compound (2E)-1-(R phenyl)-3-(dimethylamino)prop-2-en-1-one , (R) is one of the derivatives used for acetophenone (R = Br, NH_2). The second step, treatment of benzaldehyde derivatives with hydrazine to prepare the second compound in the series (1E) - (R benzylidene) hydrazine, where (R) is one of the derivatives used for benzaldehyde to get the Shiff bases. And the third step, treatment of the first step products with the products of the second step. Each product from the first step gives us a series of Pyrazole compounds with its reaction with products of the second step interaction after the other. Thus, we can bring a number of compounds, the sum of the first step products multiplied by the products of the second step. The third-step reaction is a ring-blocking reaction to form a combined heterogeneous ring of Pyrazole. Finally, characterization these compounds with infrared spectra, NMR spectrum and mass spectrometry.

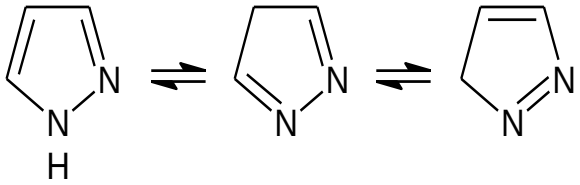
Introduction

Pyrazole, which is five-membered with two-nitrogen-containing heterocycle, are critical natural mixes for pharmaceutical [1] and agrochemical industry [2]. Various mixes containing pyrazole moiety are known to display hostile to hyperglycemic [3], pain relieving [4], mitigating[5], antipyretic[6], antibacterial[7], antimicrobial[8], antihypertensive[9], and upper exercises[10]. They are additionally utilized as herbicides [11] and dyestuffs [12].



Structure of pyrazole.

Pyrazole is aromatic molecule because of their planar conjugated ring structures with six delocalized π-electrons. Along these lines, numerous essential properties of these particles were broke down by contrasting and the properties of derivatives of benzene [13]. Like other nitrogen including heterocycles, diverse tautomeric structures can be composed for pyrazoles. As appeared in, unsubstituted pyrazole can be spoken to in three tautomeric shapes [14]. (Scheme 1)



(Scheme 1): Tautomeric forms of unsubstituted pyrazole.

For the derivatives of pyrazole in which two carbon iotas neighboring the nitrogen particles on the

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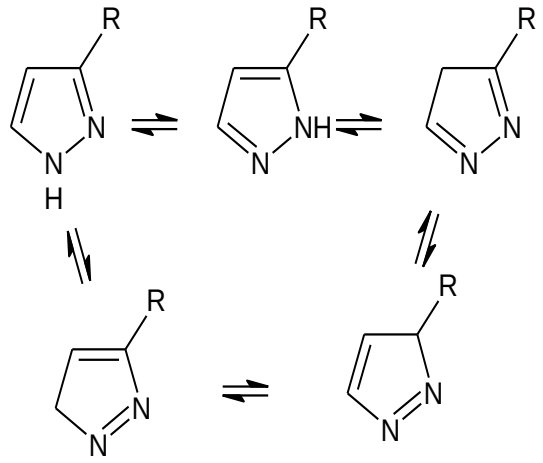
ring have distinctive substituents, five tautomeric structures are conceivable. (Scheme 2)



(Scheme 2): Five tautomeric forms of a substituted pyrazole derivative.

The term Pyrazole was given by Ludwig Knorr in 1883. Pyrazole refers to the class of simple aromatic ring organic compounds of the heterocyclic series characterized by a 5-membered ring structure composed of three carbon atoms and two nitrogen atoms in adjacent positions. Being so composed and having pharmacological effects on humans, they are classified as alkaloids, although they are rare in nature. In 1959, the first natural pyrazole, 1-pyrazolyl-alanine, was isolated from seeds of watermelons [15].

Derivatives of pyrazole have a lengthy history of utilization in agrochemicals and pharmaceutical industry as herbicides and dynamic pharmaceuticals. The current achievement of pyrazole COX-2 inhibitor has additionally featured the significance of these heterocyclic rings in therapeutic science.

An efficient examination of this class of heterocyclic lead uncovered that pyrazole containing pharmacophore dynamic specialists assume essential part in medicinal chemistry. The commonness of pyrazole centers in naturally dynamic particles has empowered the requirement for exquisite and effective approaches to make these heterocyclic lead [15].

Schiff bases:

Schiff bases are a gathering of natural intermediates, which are frequently utilized as a part of the union and concoction investigation. Schiff bases are buildup results of essential amines with carbonyl mixes picking up significance step by step in intrcophore for design and development of various bioactive lead compounds. Synthesis Pyrazole derivatives by close the ring from acetophenone derivatives with benzaldehydehydrazine derivatives. The reaction of some derivatives of benzaldehydehydrazine with derivatives of acetophenone to form derivatives of Pyrazol.

Target of Research:

Synthesis new derivatives of Pyrazol.

Experimental

The following equipment and apparatus and analytical instruments were used throughout the present study by following the manufacturer instruction:

1. Melting Points were determined in open capillary tubes using model DMP-100 from China in the University of Fallujah.
2. FT-IR spectra were recorded by Fourier Transform Infra-Red spectrophotometer model Testscan Shimadzu model 8000, Japan. The recorded spectra were obtained within the range 400 -4000 cm^{1-} by using KBr disc in Baghdad University.
3. Mass spectra were recorded by GC-MS model GCMS-QP2010 SE from Shimadzu, Japan. Direct inlet of the sample was used for all the compounds in Mustansiriyah University.
4. ^{1}H NMR, ^{13}C NMR spectra were recorded by Broker spectrometer model Ultra-Shield at 300 MHz using Acetone as a solvent for all samples with TMS as internal reference standard, in al-Bayt university, Jordan**.**

Step 1: Synthesis of starting materials

Synthesis of chalcones

Synthesis (a): 1-(4-Bromophenyl)-3-(dimethylamino)prop-2-en-1-one

A solution of p-bromoacetophenone (A) (0.01 mole, 2g) and sodium hydroxide (2.4g) in 50% aqueous ethanol was prepared in open beaker (100ml). The solution was cooled down to (0-5 ^{0}C) by immersing in crushed ice path, then N,N-dimethylformamide dimethylacetal (F) (1.95g) was add to the solution gradually with continuous vigorous stirring. Then transfer to the reflux, the reaction mixture was constantly stirred for (10 hrs) with out heating, with maintaining the reaction temperature at (25±2 ^{0}C), and then ref was refluxed for (1 hr). The solvent was removed where by a solid product was obtained. The solid was wished with ice water until the wishing water is neutral, then purified by recrystallization from (methanol-ethanol 1:1) mixed solvent and dried at (25-35 ^{0}C). Other synthesis (b) where done by fallowing the same procedure, (Scheme 3). [16]

Table 1: chalcones are prepared

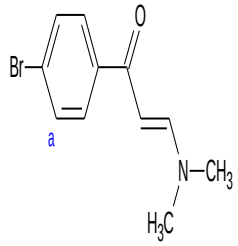
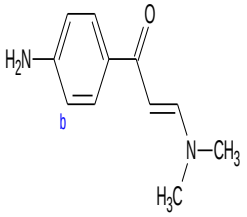
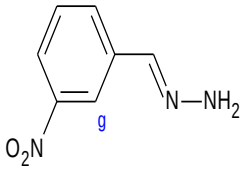
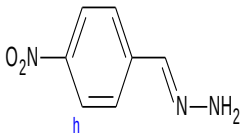
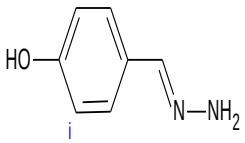
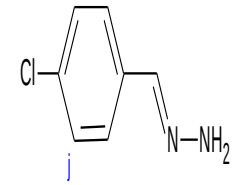
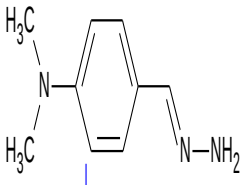
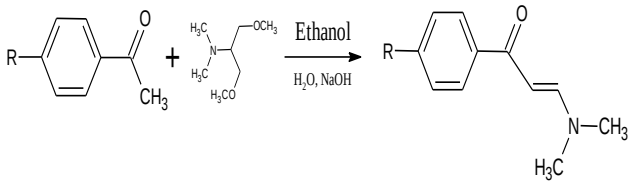
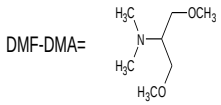
#	Compounds	Chemical formula	M. Wt.	IUPAC Name	Color	m. p C^0	%
a		$C_{11}H_{12}BrNO$	254.12	1-(4-Bromophenyl)-3-(dimethylamino)prop-2-en-1-one	Golden	88	71
b		$C_{11}H_{14}N_2O$	190.24	1-(4-Aminophenyl)-3-(dimethylamino)prop-2-en-1-one	yellow	94	75

Table 2: Schiffs' bases (imines) are prepared

#	Compounds	Chemical formula	M. Wt.	IUPAC Name	Color	m. p C^0	%
g		$C_7H_7N_3O_2$	165.15	(3-nitrobenzylidene) hydrazine	yellow	122	82
h		$C_7H_7N_3O_2$	165.15	(4-nitrobenzylidene) hydrazine	yellow	143	88
i		$C_7H_8N_2O$	136.15	(4-Hydroxybenzylidene) hydrazine	yellow	118	80
j		$C_7H_7ClN_2$	154.59	(4-Chlorobenzylidene) hydrazine	yellow	136	79
l		$C_9H_{13}N_3$	163.22	4[hydrazinylidenemethyl]-N,N-dimethylaniline	yellow	110	83





R= Br , NH_2

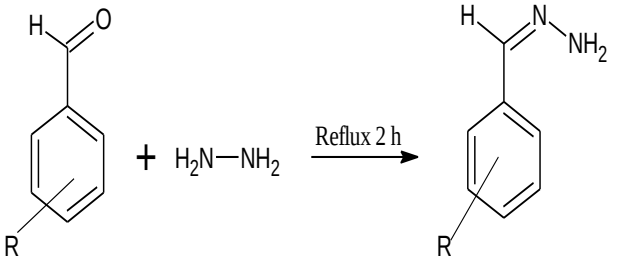
DMF-DMA = N,N-dimethylformamide dimethyl acetal

(Scheme 3)

Step2: Synthesis of Schiffs' bases (imines)

Synthesis (h): (4-nitrobenzylidene)hydrazine

A reaction mixture of 4-nitrobenzaldehyde (H) (0.01mol, 2g) and hydrazine (88%) (N) (2.7ml) in absolute ethanol (10ml) was prepared and place in a round bottom flask (100ml), equipped with reflux condenser and magnetic stirring bar. The reaction mixture was refluxed for (2hrs) and then left to cool down in an ice bath where upon a solid product separated out. The product was recrystallized from ethanol and dried at 70^0C. All other synthesis (g-l) were down by following the same procedure, (Scheme 4). [17]



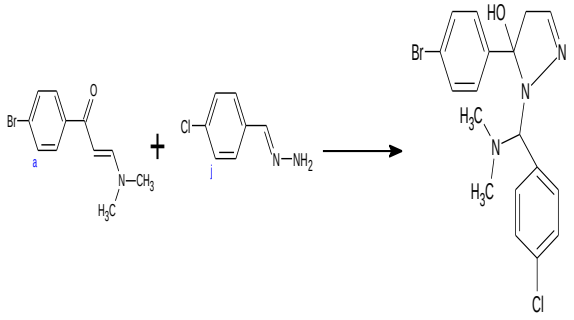
R- benzylidenehydrazine

R= NO_2,Cl,NMe_2,OH

(Scheme 4)

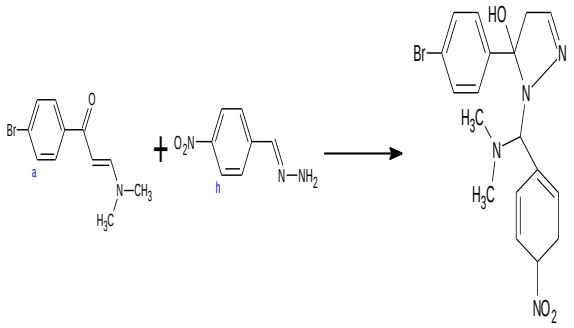
Step 3: Synthesis of Pyrazol Derivatives

-Synthesis (S1) 5-(4-bromophenyl)-1-[(4-chlorophenyl)(dimethylamin-o)methyl]-4,5-dihydro-1H-pyrazol-5-ol from (a) 1-(4-bromophenyl)-3-(dimethylamino)prop-2-en-1-one with (j) (4-chlorobenzylidene)- hydrazine. After dissolve 0.001mol, 0.4 g of (a) in ethanol and dissolve 0.3 g of (j) in ethanol also but the process of dissolving must be complete and mix the solution very well and reflux 2 hrs. We let the solution be cold and treat it in a snowy bath or treat it by acid and a piece of ice. We observe the appearance of the deposit [16].



S1

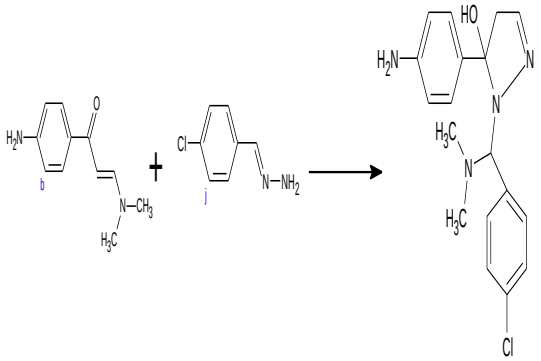
-Synthesis (S2) 5-(4-bromophenyl)-1-[(dimethylamino)(4-nitrophenyl)me-thyl]-4,5-dihydro-1H-pyrazol-5-ol from (a) 1-(4-bromophenyl)-3-(dimethylamino)prop-2-en-1-one with (h) (4-nitrobenzylidene)- hydrazine. After dissolve 0.001mole, 0.4 g of (a) in ethanol and dissolve 0.3 g of (h) in ethanol also but the process of dissolving must be complete and mix the solution very well and reflux 2 hrs. We let the solution be cold and treat it in a snowy bath or treat it by acid and a piece of ice. We observe the appearance of the deposit.



S2

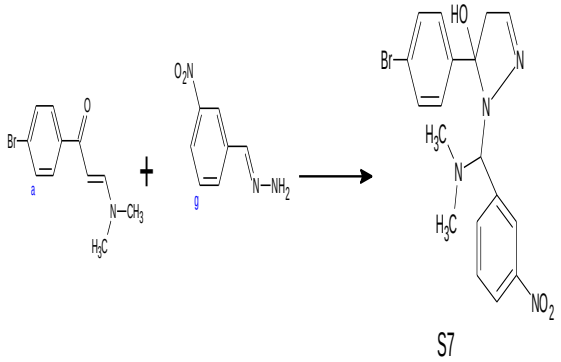
-Synthesis (S5) 5-(4-aminophenyl)-1-[(4-chlorophenyl)(dimethylamino)m-ethyl]-4,5-dihydro-1H-pyrazol-5-ol from (b) 1-(4-aminophenyl)-3-

(dimethylamino)prop-2-en-1-one with (j) (4-chlorobenzylidene)- hydrazine. After dissolve 0.002mol, 0.4 g of (b) in ethanol and dissolve 0.3 g of (j) in ethanol also but the process of dissolving must be complete and mix the solution very well and reflux 2 hrs. We let the solution be cold and treat it in a snowy bath or treat it by acid and a piece of ice. We observe the appearance of the deposit.



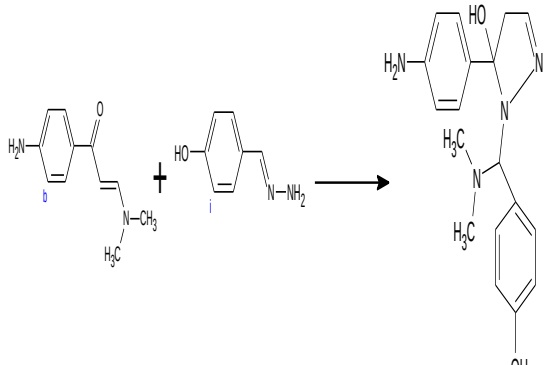
S5

-Synthesis (S7) 5-(4-bromophenyl)-1-[(3-nitrophenyl)(dimethylamin-o)methyl]-4,5-dihydro-1H-pyrazol-5-ol from (a) 1-(4-bromophenyl)-3-(dimethylamino)prop-2-en-1-one with (g)(3-nitrobenzylidene)- hydrazine. After dissolve 0.001mol, 0.4 g of (a) in ethanol and dissolve 0.3 g of (g) in ethanol also but the process of dissolving must be complete and mix the solution very well and reflux 2 hrs. We let the solution be cold and treat it in a snowy bath or treat it by acid and a piece of ice. We observe the appearance of the deposit.



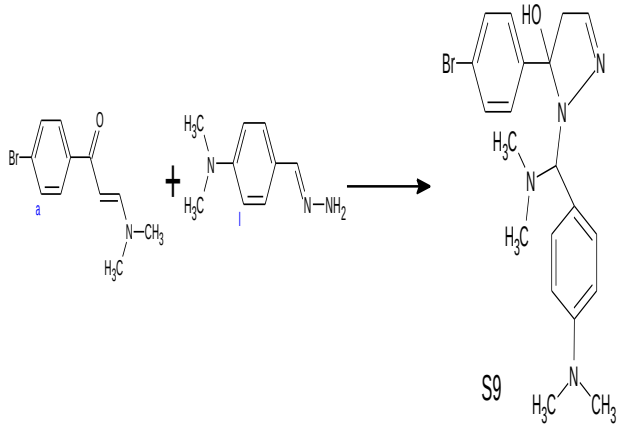
S7

-Synthesis (S8) 5-(4-aminophenyl)-1-[(4-hydroxyphenyl)(dimethylamin-o)methyl]-4,5-dihydro-1H-pyrazol-5-ol from (b) 1-(4-aminophenyl)-3-(dimethylamino)prop-2-en-1-one with (i) (4-hydroxybenzylidene)- hydrazine. After dissolve 0.002mol, 0.4 g of (b) in ethanol and dissolve 0.3 g of (i) in ethanol also but the process of dissolving must be complete and mix the solution very well and reflux 2 hrs. We let the solution be cold and treat it in a snowy bath or treat it by acid and a piece of ice. We observe the appearance of the deposit.



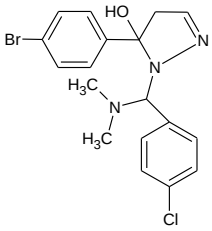
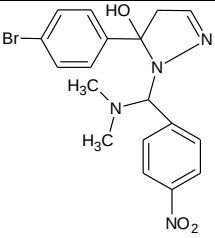
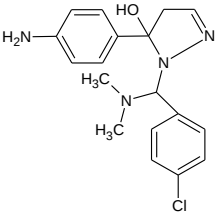
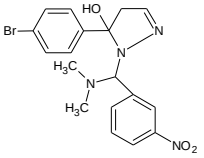
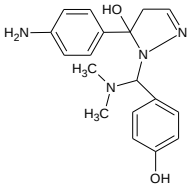
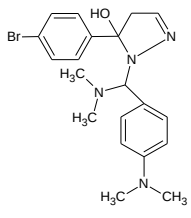
S8

-Synthesis (S9) 5-(4-bromophenyl)-1-[(4-dimethylaminephenyl)(dimethyl-amino)methyl]-4,5-dihydro-1H-pyrazol-5-ol from (a) 1-(4-bromophenyl)-3-(dimethylamino)prop-2-en-1-one with (l) 4-[hydrazinylidenemethyl]-N,N-dimethylaniline. After dissolve 0.001mol, 0.4 g of (a) in ethanol and dissolve 0.3 g of (l) in ethanol also but the process of dissolving must be complete and mix the solution very well and reflux 2 hrs. We let the solution be cold and treat it in a snowy bath or treat it by acid and a piece of ice. We observe the appearance of the deposit.



S9

Table 3: Pyrazol Derivatives are prepared

#	Compounds	Chemical formula	M. Wt.	IUPAC Name	Color
S1		$C_{18}H_{19}BrClN_3O$	408.72	5-(4-bromophenyl)-1-[(4-chlorophenyl)(dimethylamino)methyl]-4,5-dihydro-1H-pyrazol-5-ol	yellow
S2		$C_{18}H_{19}BrN_4O_3$	419.27	5-(4-bromophenyl)-1-[(dimethylamino)(4-nitrophenyl)me-thyl]-4,5-dihydro-1H-pyrazol-5-ol	yellow
S5		$C_{18}H_{21}ClN_4O$	344.84	5-(4-aminophenyl)-1-[(4-chlorophenyl)(dimethylamino)m-ethyl]-4,5-dihydro-1H-pyrazol-5-ol	brown
S7		$C_{18}H_{19}BrN_4O_3$	419.27	5-(4-bromophenyl)-1-[(3-nitrophenyl)(dimethylamino)methyl]-4,5-dihydro-1H-pyrazol-5-ol	yellow
S8		$C_{18}H_{22}N_4O_2$	326.39	5-(4-aminophenyl)-1-[(4-hydroxyphenyl)(dimethylamino)methyl]-4,5-dihydro-1H-pyrazol-5-ol	yellow
S9		$C_{20}H_{25}BrN_4O$	417.34	5-(4-bromophenyl)-1-[(4-dimethylaminephenyl)(dimethylamino)methyl]-4,5-dihydro-1H-pyrazol-5-ol	orange

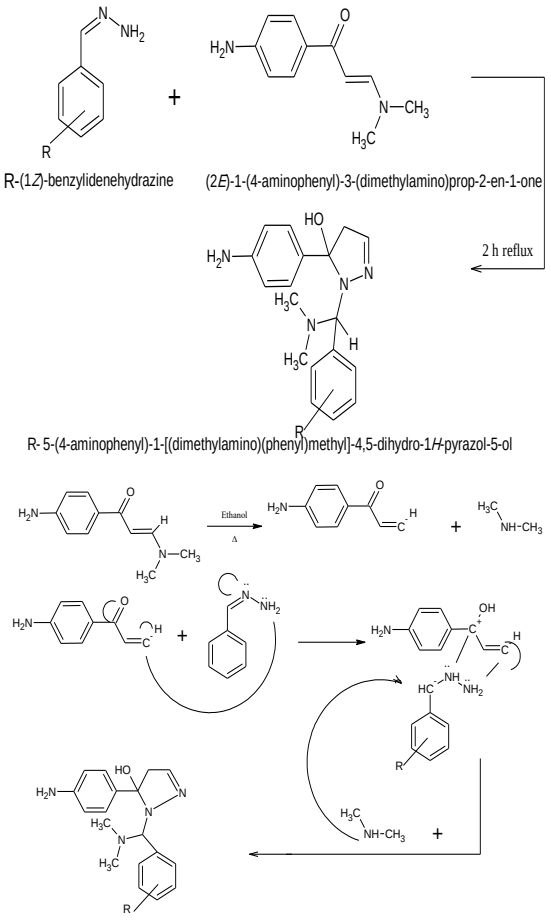
Results and discussion

This study consisted of one part includes the preparation of new derivatives of Pyrazole performing their identification by the available spectroscopic, analytical and chemical methods. Beginning react acetophenone derivatives with DMF-DMA to form the Chalcones. The second step react derivatives of benzaldehyde with hydrazine to form Schiff bases. The basis of the study is based on the synthesis of Pyrazole from a new way through close the ring by product of Chalcone and product of Schiff base. After many experiments in the laboratory, we found the vehicles according to the research, then samples were sent for identify them.

After observing the charts for FT-IR, we see disappearance peak of carbonyl and appearance peak of (OH), that is, we got the expected results in principle. The value of the base peak is apparent in chart of Mass spectrum Confirm that we have obtained the required results.

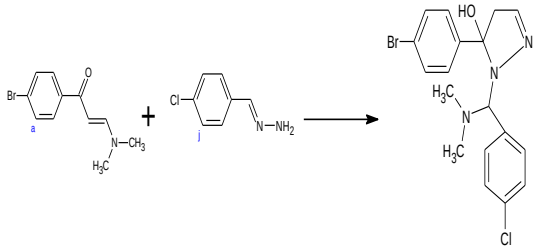
The following are listed compounds which were prepared with their equations and their identification and their charts of FT-IR, HNMR, C^{13}NMR and Mass to each other.

The target compounds, 1,5,5-trisubstituted-4,5-dihydro-1-H-pyrazol were efficiently synthesis with good high (50-80%). The reaction proceeds via nucleophilic cycloaddition reaction between the synthesized chalcones acd Schiff base in absolute ethanol at reflux condition, which bring about a fruitful attack to form an intermediate compounds which collapses in many steps to give the target product. The most likely plausible mechanism for the formation of the product may be depicted by (Scheme 5)



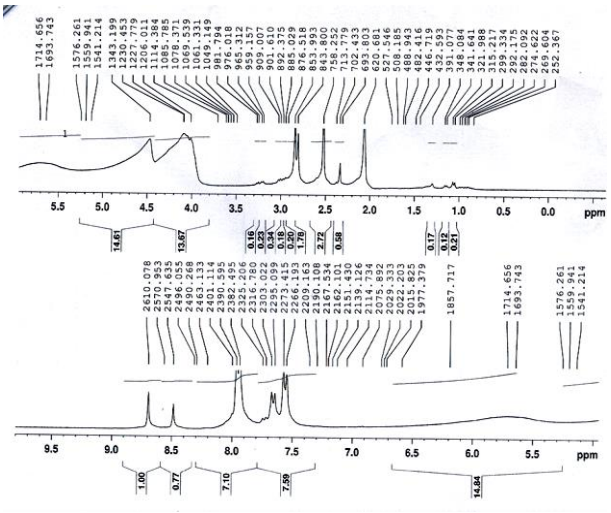
(Scheme5)

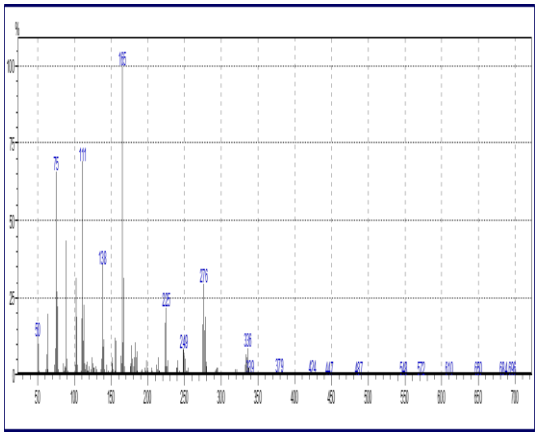
5-(4-bromophenyl)-1-[(4-chlorophenyl)(dimethylamin-o)methyl]-4,5-dihydro-1H-pyrazol-5-ol:[S1]



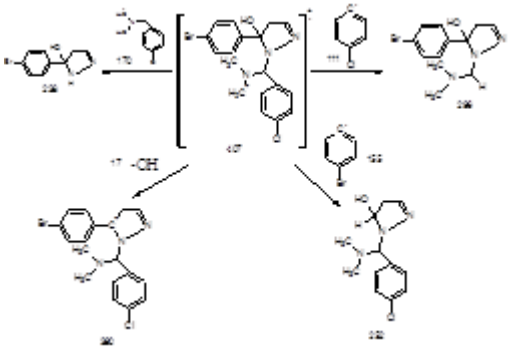
S1

As light yellow crystals yielded by (59%), m.p 145°C (from Acetone); IR (KBr) ν_{max}/cm^{-1}: 3458 (OH), 3047 (=CH), 2941 (-CH), 1623 (C=C), 1589 (Ar-H), 821 (C-H Ar), 501 (C-Br), 775 (C-Cl); ^{1}H-NMR (Acetone): δ 4.10 (s, 1H, OH), 7.60 (m, 8H, Ar-H), 8.49 (s, 1H, CH=N$^+$), 1.10 (d, 2H, CH_2CH), 2.30 (s, 1H, Ar-C); MS *m/z* (%): 409 (M^+, 0.30), 394 (0.25), 379 (0.50), 249 (12), 225 (22), 183 (16), 165 (100), 111 (73), 75 (67). Anal. Calcd. For $C_{18}H_{19}BrClN_3O$ (408.72): C (52.90%), H (4.69%), Br (19.55%), Cl (8.67%), N (10.28%), O (3.91%)(Scheme 6). [18], [19], [20]

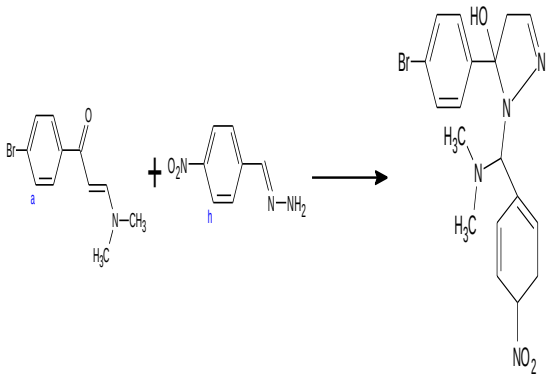




(Scheme 6): Mass spectrometer and the spectrum HNMR for S1

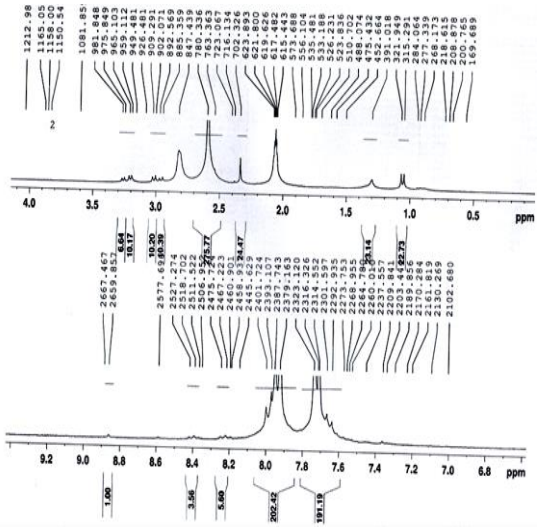


5-(4-bromophenyl)-1-[(dimethylamino)(4-nitrophenyl)me-thyl]-4,5-dihydro-1H-pyrazol-5-ol:[S2]



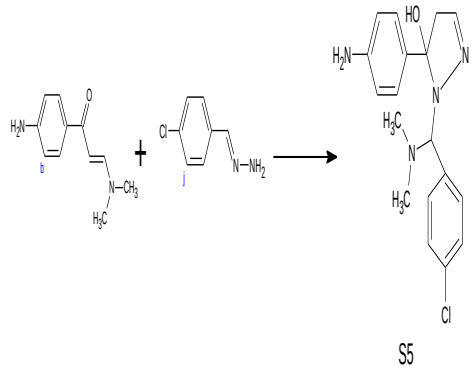
S2

As yellow crystals yielded by (66%), m.p 217ºC (from Acetone); IR (KBr) ν_{max}/cm^{-1}: 3425 (OH), 3074 (=CH), 2927 (-CH), 1683 (C=C), 1523 (Ar-H), 1487 (CH_2), 1346 (NO_2), 684 (C-Br), 1394 (CH_3); ^{1}H-NMR (Acetone): δ 8.6 (s, 1H, CH=N^+), 2.90 (s, 1H, OH), 7.70 (m, 8H, ArH), 7.90 (d, 2H, Ar-H), 2.10 (s, 1H, $CHNR_2$), 1.30 (d, 2H, CH_2CH), 1.10 (t, 3H, $CHCH_2$); ^{13}C-NMR (Acetone): δ 25.75 (C-Br), 34.16 (C-N), 130 (Ar-H), 131 (Ar-H), 127 (=CR2), 60 (C-O), 28.11, 28.41, 28.61, 28.91, 29.11, 29.41, 29.61 (C-CH_2). Anal. Calcd. For $C_{18}H_{19}BrN_4O_3$ (419.27): C (51.56%), H (4.57%), Br (19.06%), N (13.36%), O (11.45%).



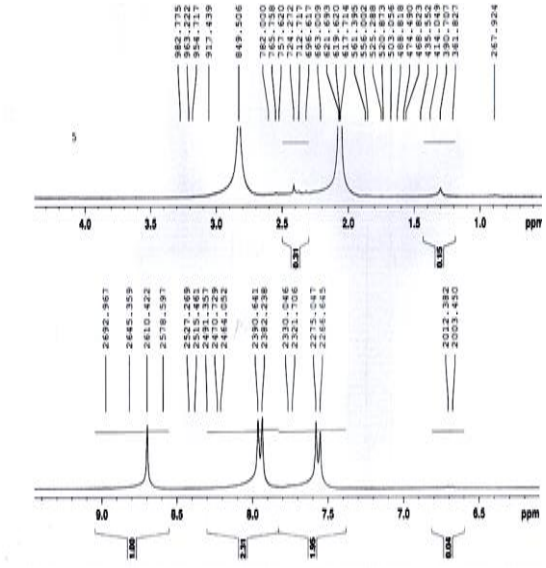
(Scheme 7): the spectrum HNMR for S2

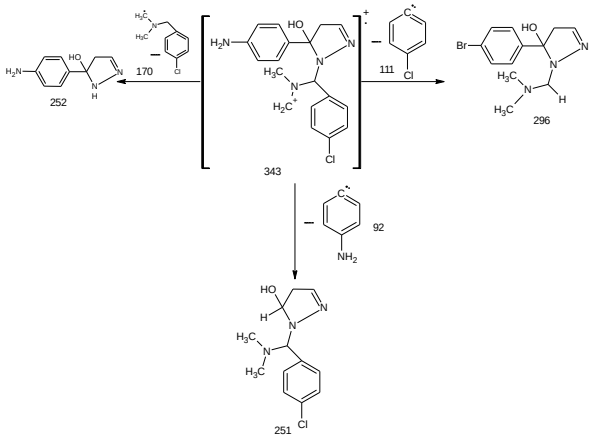
5-(4-aminophenyl)-1-[(4-chlorophenyl)(dimethylamino)m-ethyl]-4,5-dihydro-1H-pyrazol-5-ol:[S5]

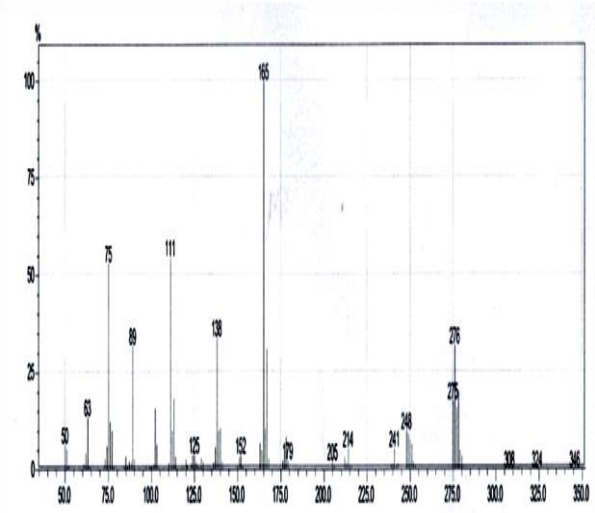


S5

As brown crystals yielded by (70%), m.p 213ºC (from Acetone); IR (KBr) ν_{max}/cm^{-1}: 3434 (OH), 3320,3340 (NH_2), 3049 (=CH), 2941 (-CH), 1623 (C=C), 1487 (Ar-H), 1087 (C-O), 819 (C-Cl); ^{1}H-NMR (Acetone): δ 2.80 (s, 1H, OH), 7.60 (m, 8H, Ar-H), 7.90 (d, 2H, Ar-H), 8.70 (s, 1H,CH=N^+), 1.30 (d, 2H, CH_2CH), 2.10 (t, 3H,$CHCH_2$), 2.40 (s, 1H, Ar-C); MS *m/z* (%): 347 (M^+, 1.30), 308 (1.90), 248 (17), 165 (100), 111 (60), 251 (5) Anal. Calcd. For $C_{18}H_{21}ClN_4O$ (344.84): C (62.69%), H (6.14%), Cl (10.28%), N (16.25%), O (4.64%).(Scheme7)

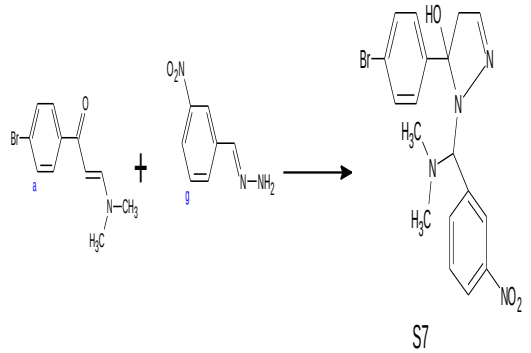






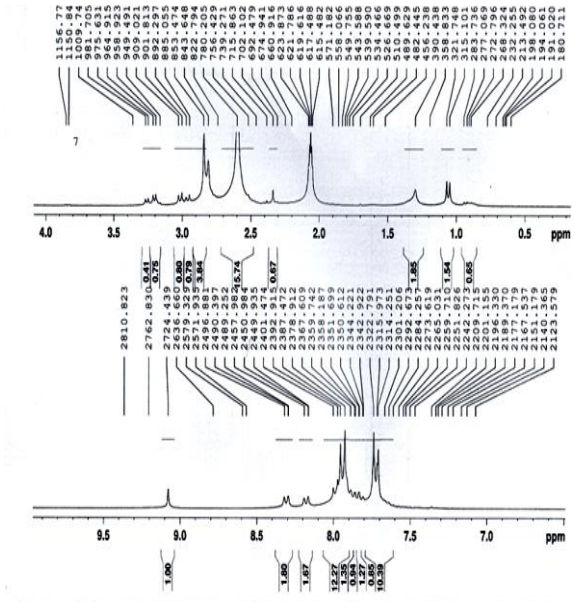
(Scheme 8): Mass spectrometer and the spectrum HNMR for S5

5-(4-bromophenyl)-1-[(3-nitrophenyl)(dimethylamin-o)methyl]-4,5-dihydro-1H-pyrazol-5-ol:[S7]



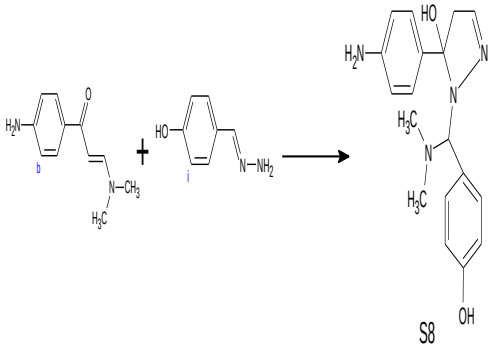
S7

As yellow crystals yielded by (69%), m.p 167°C (from Acetone); IR (KBr) ν_{max}/cm^{-1}: 3298 (OH), 2854 (-CH), 1683 (C=C), 1523 (Ar-H), 1342 (NO_2), 1201 (C-O), 700 (C-Br); ^{1}H-NMR (Acetone): δ 2.60 (s, 1H, OH), 7.50 (m, 8H, Ar-H), 7.90 (m, 8H, Ar-H), 2.90 (s, 1H, $CHNR_2$), 1.10 (d, 2H, CH_2CH), 2.10 (t, 3H, $CHCH_2$), 2.30 (s, 1H, Ar-C). Anal. Calcd. For $C_{18}H_{19}BrN_4O_3$ (419.27): C (51.56%), H (4.57%), Br (19.06%), N (13.36%), O (11.45%).



(Scheme 9): the spectrum HNMR for S7

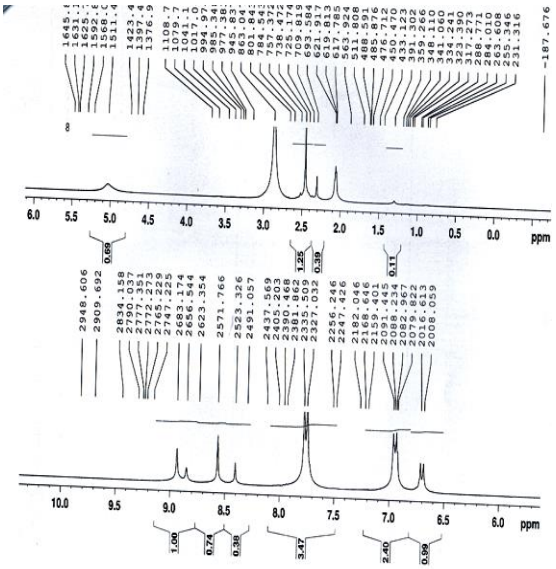
5-(4-aminophenyl)-1-[(4-hydroxyphenyl)(dimethylamin-o)methyl]-4,5-dihydro-1H-pyrazol-5-ol:[S8]



S8

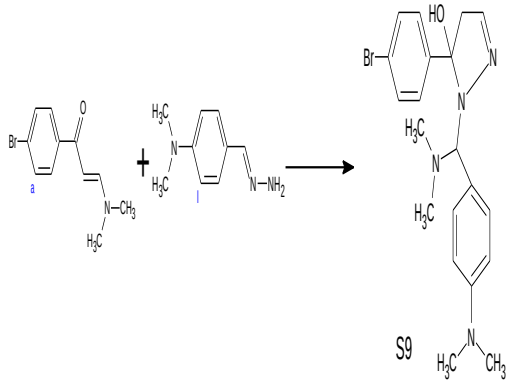
As yellow crystals yielded by (73%), m.p 185°C (from Acetone); IR (KBr) ν_{max}/cm^{-1}: 3398 (OH), 3334,3320 (NH_2), 3064 (=CH), 2943 (-CH), 1604 (C=C), 1514 (Ar-H), 1169 (C-O); ^{1}H-NMR (Acetone): δ 6.95 (m, 8H, Ar-H), 7.70 (m, 8H, Ar-H), 2.10 (s, 1H, $CHNR_2$), 1.30 (d, 2H, CH_2CH), 8.60 (s, 1H, $CH=N^+$), 6.70 (s, 1H, ArOH); ^{13}C-NMR (Acetone): δ 13.40 (R_2CH_2), 127 (Ar-H), 128 (Ar-H), 130 (Ar-H), 113 (ArOH), 116 (OH), 160 (Ar-NH_2), 28.11, 28.41, 28.61, 28.91, 29.11, 29.41, 29.61 (C-CH_2). Anal. Calcd. For

$C_{18}H_{22}N_4O_2$ (326.39): C (66.24%), H (6.79%), N (17.17%), O (9.80%).

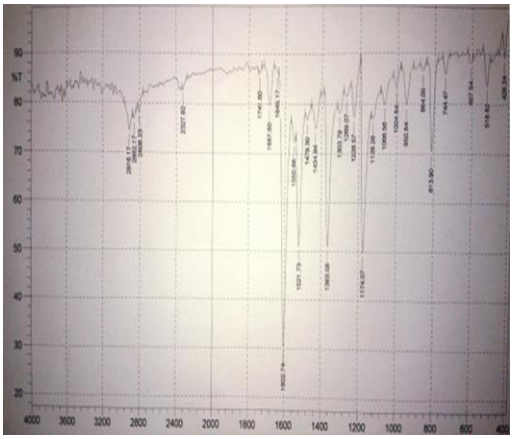


(Scheme 10): the spectrum HNMR for S8

5-(4-bromophenyl)-1-[(4-dimethylaminephenyl)(dimethylamino)methyl]-4,5-dihydro-1H-pyrazol-5-ol:[S9]



As orange crystals yielded by (57%), m.p 177°C (from 1:1 Ethanol + Methanol); IR (KBr) ν_{max}/cm^{-1}: 3200 (OH), 2916 (-CH), 1602 (C=C), 1521 (Ar-H), 1363 (CH_3), 1174 (C-OH). Anal. Calcd. For $C_{20}H_{25}BrN_4O$ (417.34): C (57.56%), H (6.04%), Br (19.15%), N (13.42%), O (3.83%).



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تحضير وتشخيص بعض المركبات الحلقية الغير متجانسة المحتوية على للبايرازول

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الخلاصة :

تتضمن هذه الدراسة تحضير بعض المركبات الحلقية الغير متجانسة المدمجة للبايرازول ، ويتم العمل بثلاث خطوات . الخطوة الاولى ، معاملة مشتقات الاسيتوفينون مع ال (DMF-DMA) لتحضير المركب الاول 1-(R phenyl)-3-(dimethylamino)prop-2-en-1-one

حيث ان (R) هو احد المشتقات المستخدمة للاسيتوفينون . الخطوة الثانية ، معاملة مشتقات البنزلدهايد مع الهيدرازين لتحضير المركب الثاني في السلسلة (R benzylidene) hydrazine حيـــث ان (R) هو احد المشتقات المستخدمة للبنزلدهايد للحصول على قواعد شف .

الخطوة الثالثة ، معاملة نواتج الخطوة الاولى مع نواتج الخطوة الثانية ، ليعطينا كل ناتج من الخطوة الاولى سلسلة من مركبات البايزول بمفاعلته مع نواتج الخطوة الثانية تفاعل بعد الاخر ، وبذلك باستطاعتنا ان نحضر عدد من المركبات مجموعها نواتج الخطوة الاولى مضروب بنواتج الخطوة الثانية . يكون تفاعل الخطوة الثالثة تفاعل غلق للحلقة لتكوين مركبات حلقية غير متجانسة مدمجة للبايرزول . واخيرا ، تشخيص هذه المركبات بطيف الاشعة تحت الحمراء وطيف الرنين النووي المغناطيسي وطيف الكتلة .