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*Synthesis and characterization of new benzotriazole derivatives

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Abstract

This research involves synthesis of some derivatives of benzotriazole and new heterocyclic derivatives such as (pyrazoline, pyrimidine, benzopyran) which have chemical and biological importance.

The first step, include the synthesis of 1-N-Acetyl-benzotriazole from reaction benzotriazole with acetic anhydride in presence of some drops of sulfuric acid.

The second step, include synthesis the a series of α,β-unsaturated carbonyl compounds (chalcones) ,by consdensation of 1-N-Acetyl-benzotriazole ,with different aromatic aldehydes[4-(dimethylamino)benzaldehyde,4-hydroxybenzaldehyde,4-nitrobenzaldehyde,4-clorobenzal dehyde] to give new chalcone (A ,B ,C ,D) respectively.

A. 1-N (3-(4-(dimethylamino phenyl)propenone) Benzotriazole.
B. 1-N (3-(4-hydroxyphenyl) propenone) Benzotriazole.
C. 1-N (3-(4-nitrophenyl) propenone) Benzotriazole.
D. 1-N (3-(4-chlorophenyl) propenone) Benzotriazole.

The three step, include the synthesis the A series of different heterocyclic.

These compounds (chalcones) were used as precursors in preparation of a number of cyclic organic compounds through their reaction with :

1- hydrazine, Phenyl hydrazine in methanoic acid to form another series of pyrazoline compounds (A1,B1,C1,D1, A2,B2,C2,D2) .
2- thiocemecarbezid to form pyrazoline derivatives compounds (A3,B3,C3,D3).

3- uear,thiouear and guaniden to from pyrimidine derivatives compounds (A4,A5,A6,B4,B5,B6,C4,C5,C6,D4,D5,D6).

4- Resorcinol in Hydrochloric acid to form benzopyran derivatives compounds (A7,B7,C7,D7) .

Key world :benztriazole, chalcone, pyrazoline, pyrimidine,

***The Research is apart of on MSC. Thesis in the case of the First researcher**

Introduction.

Heterocyclic compounds occur widely in nature and in a variety of non-naturally occurring compounds. A large number of heterocyclic compounds are essential to life. Various compounds such as alkaloids, antibiotics, essential amino acids, vitamins, haemoglobin, hormones and a large number of synthetic drugs and dyes contain heterocyclic ring systems. Heterocycles play a vital role in pharmacological, agricultural and synthetic fields. There is a large number of synthetic heterocyclic compounds with additional important applications and many are valuable intermediates in synthesis[1,5].

The starting benzotriazole derivatives are usually prepared from the parent heterocycle by N-alkylation with a halide, or via reaction with aldehydes or acetals,which can lead to mixtures of 1- and 2-substituted benzotriazoles, however the reactivities of the two isomers are similar. For clarity, only reactions of 1-substituted compounds[6,9].

. Considerable interest has been focused on the pyrazoline structure, which has been found to possess a borad specturm of biological activities such as tranquillizing, muscle relaxant, psychoanalytic, anticonvulsant, antihypertensive, and antidepressant activities. The discovery of modern drug development also points out the unpredictability of biological activity from structural modification of a prototype drug. Pyrimidines are one of the heterocyclic compounds containing of six membered unsaturated structure composed of two nitrogen atoms at position 1 and 3[10,14] .

Pyrimidines have been subjected to a large number of different modifications in order to obtain derivatives having different biological properties Several groups have studied the chemistry and pharmacological properties of pyrimidine derivatives[15,18] .

Chemicals and Instruments

All chemicals used were supplied from Merck , BDH and Fluke chemicals company .

Melting points were recorded using Electro thermal melting point apparatus , UK.

F.T.I.R spectra were recorded using Fourier transform infrared SHIMADZU FT.IR-8400S infrared spectrophotometer by KBr disc, University of Kufa. The elemental analysis were recorded using E.A.G.E.R.-100, Carlo Erba, Italy, measurements were made at the of,Bio Chemistry Lab, ,University of Kufa

Thin layer chromatography (TLC) was performed on aluminum plates and coated with layer of silica gel, compounds were detected by iodine vapor.^{1}H-NMR were recorded on Fourier transformation Varian spectrometer, operating at(400 MHz) with (DMSO-d_6), measurement were made at the department of chemistry, Kashan University, Iran.

General procedure for synthesis (1-N-Acetylbenzotriazole) (BTAC).

A mixture of 0.1 mol benzotriazole, 25 ml of acetic anhydride with some drops of sulfuric acid, refluxed (1 hr) at (55-60)℃ in water bath, then added into crushed ice with stirring. The precipitate formed was filtered and washed with D.W. . The progress of the reaction was monitored by TLC. (benzene: ethanol 1:1).

General procedure for synthesis chalcone.

To a stirred mixture of 0.005mol of 1-N-Acetylbenzotriazole (0.805g) and 0.005mol of aromatic aldehydes in 25 mL ethanol at room temperature, 30% NaOH aqueous solution was added portion-wise after which stirring was continued for further 3 hr. The color precipitate formed was filtered and washed with 3% aqueous HCl, and crystallized from ethanol .

General procedure for the synthesis of (pyrazoline).

To a stirred solution of chalcone (A,B,C,D 0.002 mol) in 15 ml EtOH (96 %) was added phenyl hydrazine or hydrazine Hydrate (0.002 mol) and methanoic acid (2.5 ml) at room temperature. The reaction mixture was heated to reflux for (2 hr). The progress of the reaction was monitored by TLC (ethyl acetate: methanol 8:2). The EtOH was removed under reduced pressure and residue recrystalized from EtOH.

General procedure for the synthesis (pyrazoline)from thiocemecarbezid.

A mixture of 0.001 mol of chalcones and 0.004 mol of thiocemecarbezid dissolved in 30 ml ethanol, then added 0.002 mol NaOH dissolved in 20 ml of ethanol , refluxed the mixture for (12 hr) .The progress of the reaction was monitored by TLC (ethyl acetate/hexane, 8:2). The EtOH was removed under reduced pressure and residue recrystalized from EtOH .

General procedure for synthesis of pyrimidine

A mixture of chalcone (0.0014 mol), urea , thiourea or guanidine (0.0014 mol) dissolved in absolute ethanol (20mL) and sodium ethoxide (1g Na) in ethanol absolut(10 mL) was taken in a flask and heated under reflux for (24 hr). Completion of reaction was monitored by (TLC). The reaction mixture was concentrated under vacuum. The precipitates were filtered under suction and washed with W.D. The crude product was purified by silica gel column chromatography with ethyl acetate/n-hexane.

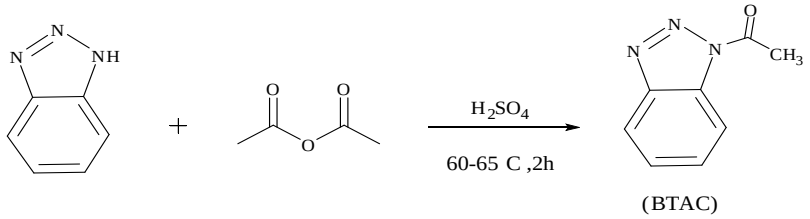
General procedure for synthesis Benzopyran (A7,B7,C7,D7).

A mixture is (0.002 mol) from one chalcones (A, B, C, D) with (0.52 gm, 0.002 mol) of resosenol, then add (2ml) of 36% hydrochloric acid (25ml) of dichloromethane , (25ml) of diethylether and refluxed for 24 hours then add water to the resulting precipitation , the product filtered and wished by ethanol and then dried and recrystallized using ethanol abs. Completion of reaction was monitored by thin layer chromatography (TLC).

Result and discussion.

Synthesis and identification of 1-N-Acetylbenzotriazole. [BTAC].

The first compound [BTAC] were prepared by reaction benzotriazole with acetic anhydride in presence of few drops of concentrated sulfuric acid . scheme[1].



(BTAC)

Scheme [1]

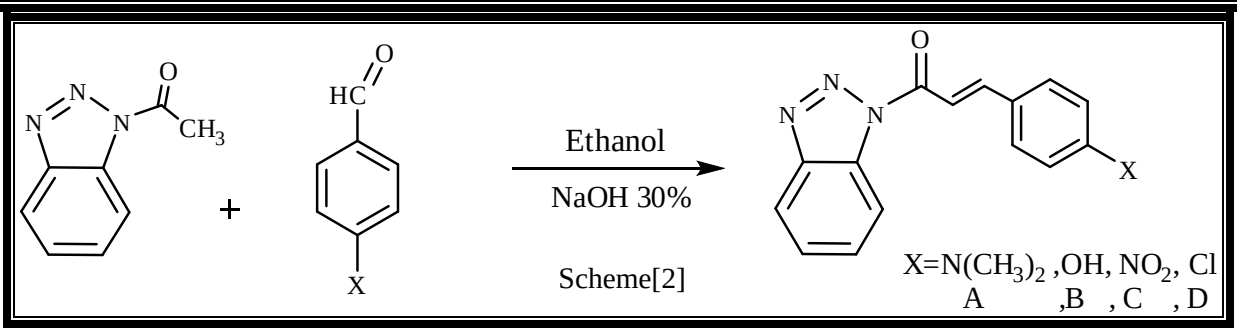
Table[1] [C.H.N]analysis,F.T.I.Rdata and some physical properties of 1-N-Acetyl-benzotriazole.

Comp. No.	formula	Calculated			M.P Č	Yield %	R_f	*Infrared data (V,cm-1) (KBr disc)*
		C%	H%	N%				
BTAC	$C_8H_7N_3O$	(59.60) 59.02	(4.38) 3.91	(26.07) 25.64	49-50	93	0.73	3095(CHar),2954(C-Hal) 1728(C=Oamid)

The F.T.I.R spectra of this compound showed disappearance of absorption band at (3375) cm^{-1} of the symmetric stretching vibration of (-NH-) group of benzotriazole and appearance band at 1728 cm^{-1} of carbonyl groups.

Synthesis and identification of chalcones [A,B,C,D].

Chalcones was chosen as a starting material for the synthesis of different heterocyclic compounds pyrazolines, pyrimidines, benzopyran derivatives by using approperative reagents for that purpose.Chalcones [A,B,C,D] are synthesized by Claisen-Schmidt condensation of 1-N-Acetylbenzotriazole and 4-substituted aromatic benzaldehyde in basic medium and ethanol as solvent to yield the desire chalcones.



Scheme[2]

The synthesized compounds were characterized by bayer test and the result was positive, that is good evidence for formatted our compounds [A,B,C,D]. The [C.H.N]analysis of these compound accepted agreement with the calculated percentages of elements were showed in the table [2].

Table [2] C.H.N analysis , F.T.I.R data and The physical properties of Chalcones.

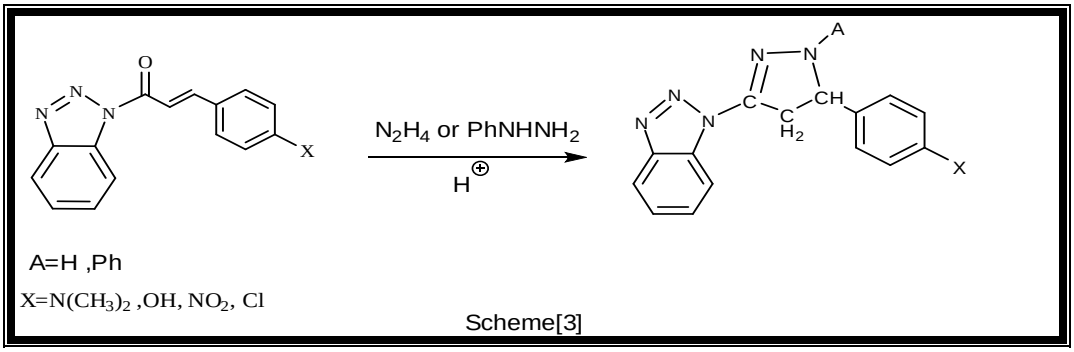
Comp. No.	formula	Calculated			M. P ^{0}C	Yield %	R_f	*Infrared data (V,cm-1) (KBr disc)*
		C%	H%	N%				
A	$C_{17}H_{16}N_4O$	(69.85) 69.36	(5.52) 5.02	(19.17) 18.58	65-66	90	0.73	3060(C-Har),3100(C-Hvinyl) 1660(C=Oamid),1600(C=C).
B	$C_{15}H_{11}N_3O_2$	(67.92) 66.98	(4.18) 3.83	(15.84) 14.95	76-77	85	0.69	3400(O-H),3057(C-Har)3050(C-Hvinyl),1665 (C=O) ,1610 (C=C)
C	$C_{15}H_{10}N_4O_3$	(61.22) 60.42	(3.43) 3.17	(19.04) 18.87	71-72	87	0.86	3060(C-Har),3118(C-Hvinyl)1661 (C=Oamid),1625(C=C),1335(NO_2) .
D	$C_{15}H_{10}ClN_3O$	(63.50) 63.01	(3.55) 2.92	(12.50) 12.03	79-80	80	0.75	2997(C-Har),3090(C-Hvinyl)1680 (C=Oamid),1610(C=C),835 (C-Cl).

The F.T.I.R spectra showed appearance of stretching vibration bands between (1550-1600)$cm^{-1}$was due to the stretching vibration of (C=C) group, with remaining absorption band at(1660-1680)$cm^{-1}$of the stretching vibration of carbonyl group (C=O). The appearance of the strong absorption band at 3450 cm^{-1} to the OH group in [B] compounds . All of these absorption bands are anther good evidence to formation [A,B,C,D].

Synthesis and identification of pyrazolines.

1. pyrazolines by reaction chalcones with hydrazine and phenyl hydrazine.

The chalcones [A,B,C,D] was further reacted with hydrazine hydrate and phenyl hydrazine in ethanol absolute to yield the corresponding pyrazoline derivatives by the following reaction.



Scheme[3]

The [C.H.N.S] analysis of synthesized compounds [A1,A2 ,B1,B2,C1,C2,D1,D2] was accepted agreement with the calculated percentage of elements were showed in table [3].

Table [3] C.H.N analysis, F.T.I.R data and The physical properties of pyrazoline.

Com. No.	formula	Calculated			M. P ^{0}C	Yield %	R_f	*Infrared data (V,cm-1) (KBr disc)*
		C%	H%	N%				
A1	$C_{17}H_{18}N_6$	-	-	-	255	90	0.6	3433(NH),3090(C-Har),2914(C-Hal) ,1600(C=C), 1550(C=N).
A2	$C_{23}H_{22}N_6$	(72.23) 72.16	(5.80) 4.87	(21.97) 20.99	115	85	0.8	3070(C-Har),2917(C-Hal), 1600(C=C), 1556(C=N).
B1	$C_{15}H_{13}N_5O$	(64.51) 64.01	(4.69) 4.12	(25.07) 24.63	230	80	0.65	3446(O-H),3396(NH),3037(C-Har),1597(C=C), 1496(C=N).
B2	$C_{21}H_{17}N_5O$	-	-	-	124	90	0.7	3440(O-H),3072(C-Har), 1600(C=C), 1560(C=N).
C1	$C_{15}H_{12}N_6O_2$	(58.44) 58.15	(3.92) 3.32	(27.26) 27.04	215	82	0.67	3408(NH),3100(C-Har), 1610(C=C), 1585 (C=N), 1344(NO_2).
C2	$C_{21}H_{16}N_6O_2$	-	-	-	146	85	0.74	3100(C-Har),1600(C=C), 1525(C=N), 1382(NO_2).
D1	$C_{15}H_{12}ClN_5$	-	-	-	240	80	0.63	3290(NH),2990(C-Har), 1586(C=C), 1530(C=N), 813(C-Cl).
D2	$C_{21}H_{16}ClN_5$	(67.47) 67.35	(4.31) 4.02	(18.73) 18.24	130	82	0.59	3055(C-Har),1600(C=C), 1556(C=N), 796(C-Cl).

The F.T.I.R spectra showed appearance of absorption band at (3440-3290) cm^{-1} of the symmetric stretching vibration of (-NH-) group of compounds[A1,B1,C1,D1] and disappearance stretching vibration bands between (1660-1680)$cm^{-1}$was due to the stretching vibration of (C=O) group,

and disappearance stretching vibration bands between (1550-1600)$cm^{-1}$was due to the stretching vibration (C=C) of vinyl group of all compounds. The remaining of the strong absorption band at 3446 cm^{-1} to the OH group in [B] compound . All of these absorption bands are anther good evidence to formation [A1,A2,B1,B2,C1,C2,D1and D2].

^{1}H-NMR spectrum (δ ppm) of (A1) showed the following characteristic chemical signals (DMSO-d_6) as a solvent, ((2H), (-C$\underline{H}_2$-)$_{Pyrazoline}$ 2.031), ((6H) (N(CH_3)$_2$ 3.031), ((1H) (-C$\underline{H}$-)$_{Pyrazoline}$ 3.992), ((1H) (-NH-) 7.418), ((Ar-H)) 6.512-8.177).

^{1}H-NMR spectrum (δ ppm) of (B2) showed the following characteristic chemical signals (DMSO-d_6) as a solvent, ((2H), (-C$\underline{H}_2$-)$_{Pyrazoline}$ 2.031), ((1H) (-C$\underline{H}$-)$_{Pyrazoline}$ 5.172), ((Ar-8H)) 6.682-7.771), ((O-H)) 8.482).

^{1}H-NMR spectrum (δ ppm), Figure [1] of (D2) showed the following characteristic chemical signals (DMSO-d_6) as a solvent, ((2H), (-C$\underline{H}_2$-)$_{Pyrazoline}$ 2.063), ((1H) (-C$\underline{H}$-)$_{Pyrazoline}$ 5.096), ((Ar-H)) 6.927-8.095).

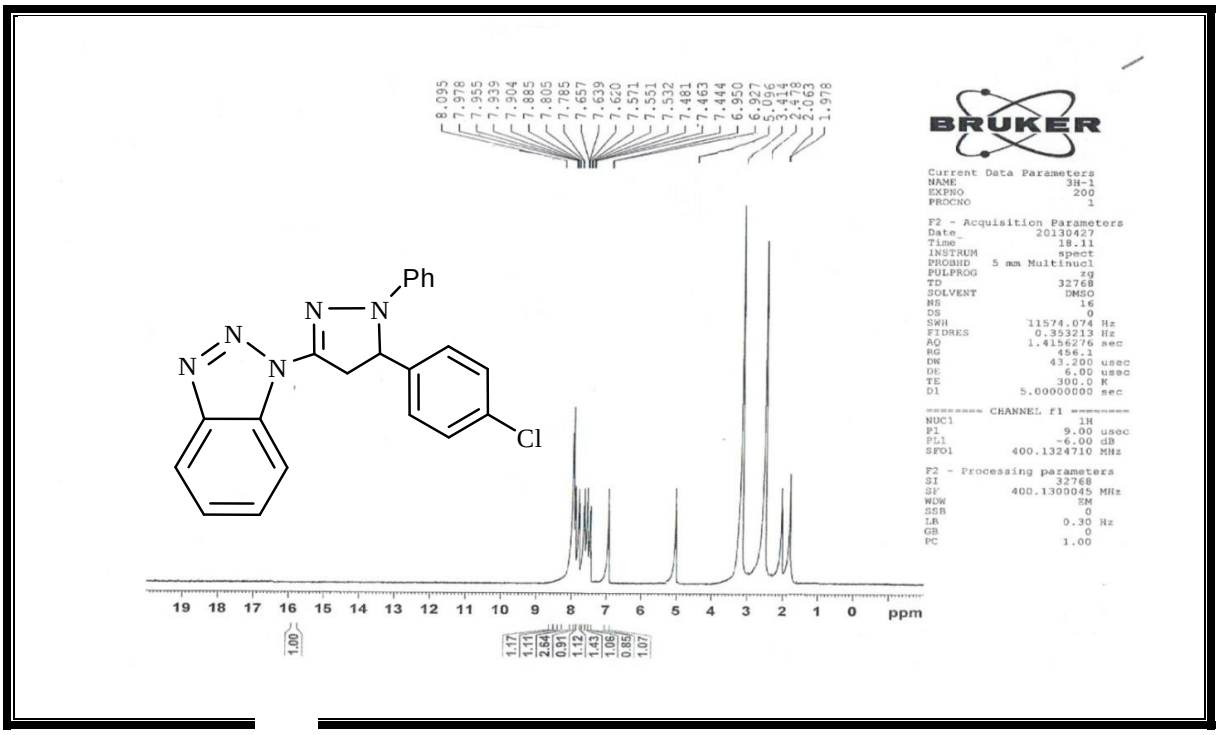
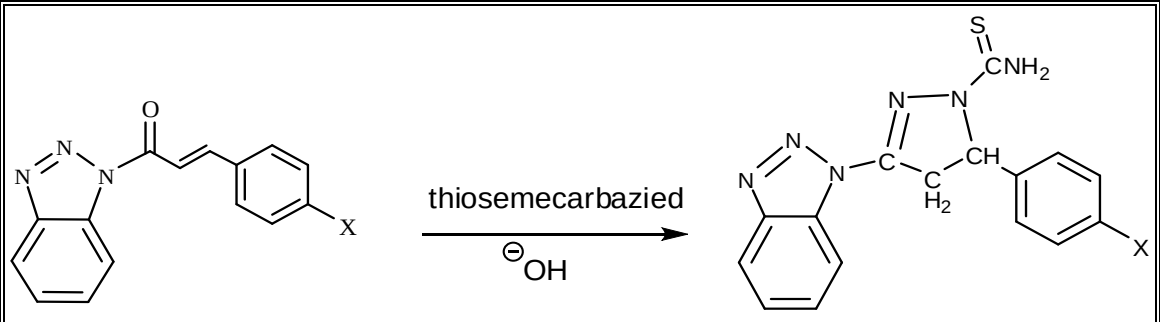


Figure [3-25] ^{1}H-NMR spectrum of the compound[D2]

2. pyrazolines by reaction chalcones with thiosemecarbazied.

The chalcones [A,B,C,D] was further reacted with thiosemecarbazied in ethanol absolute to yield the corresponding pyrazoline derivatives by the following reaction.



X=N(CH$_3$)$_2$,OH, NO$_2$, Cl

Scheme[4]

The [C.H.N.S] analysis of synthesized compounds [A3, B3 ,C3, D3] was accepted agreement with the calculated percentage of elements were showed in table [4].

Table [4] C.H.N analysis, F.T.I.R data and The physical properties of pyrazoline .

Com. No.	formula	Calculated			M. P ^{0}C	Yield %	R_f	*Infrared data (V,cm-1) (KBr disc)*
		C%	H%	N%				
A3	$C_{18}H_{19}N_7S$	(59.16) 58.67	(5.24) 4.90	(26.83) 26.31	235	83	0.63	3338,3284(NH2),3117(C-Har),2940(C-Hal),1600(C=C), 1544(C=N), 1375(C=S).
B3	$C_{16}H_{14}N_6OS$				225	85	0.7	3437(O-H),3363,3340(NH2), 3000(C-Har),1606(C=C), 1591(C=N), 1313(C=S).
C3	$C_{16}H_{13}N_7O_2S$	(52.31) 52.01	(3.57) 3.05	(26.69) 26.12	210	80	0.6	3400(NH2),3082(C-Har), 1613(C=C),1568(C=N), 1353(C=S),1325(NO_2).
D3	$C_{16}H_{13}ClN_6S$				240	90	0.58	3360,3250(NH2),3001(C-Har), 1610(C=C),1595(C=N), 1315(C=S),777(C-Cl).

The F.T.I.R spectra showed appearance of absorption two bands at (3400) cm^{-1} and (3250) cm^{-1} of the symmetric stretching vibration of (-NH_2) group and disappearance stretching vibration bands between (1660-1680)cm^{-1}was due to the stretching vibration of (C=O) group, and

disappearance stretching vibration bands between (1550-1600)$cm^{-1}$was due to the stretching vibration (C=C) of vinyl group of all compounds. The remaining of the strong absorption band at 3446 cm^{-1} to the OH group in [B3] compound . All of these absorption bands are anther good evidence to formation [A3, B3, C3and D3].^{1}H-NMR spectrum (δ ppm), Figure [2] of (C3) showed the following characteristic chemical signals (DMSO-d_6) as a solvent, ((2H), (-C$\underline{H}_2$-)$_{Pyrazoline}$ 1.914), ((1H) (-C$\underline{H}$-)$_{Pyrazoline}$ 3.701), ((Ar-8H)) 7.348-8.679), ((NH_2) 9.492).
^{1}H-NMR spectrum (δ ppm) of (D3) showed the following characteristic chemical signals (DMSO-d_6) as a solvent, ((2H), (-C$\underline{H}_2$-)$_{Pyrazoline}$ 1.858), ((1H) (-C$\underline{H}$-)$_{Pyrazoline}$ 3.701), ((Ar-H)) 6.851-7.595), ((NH_2) 9.231).

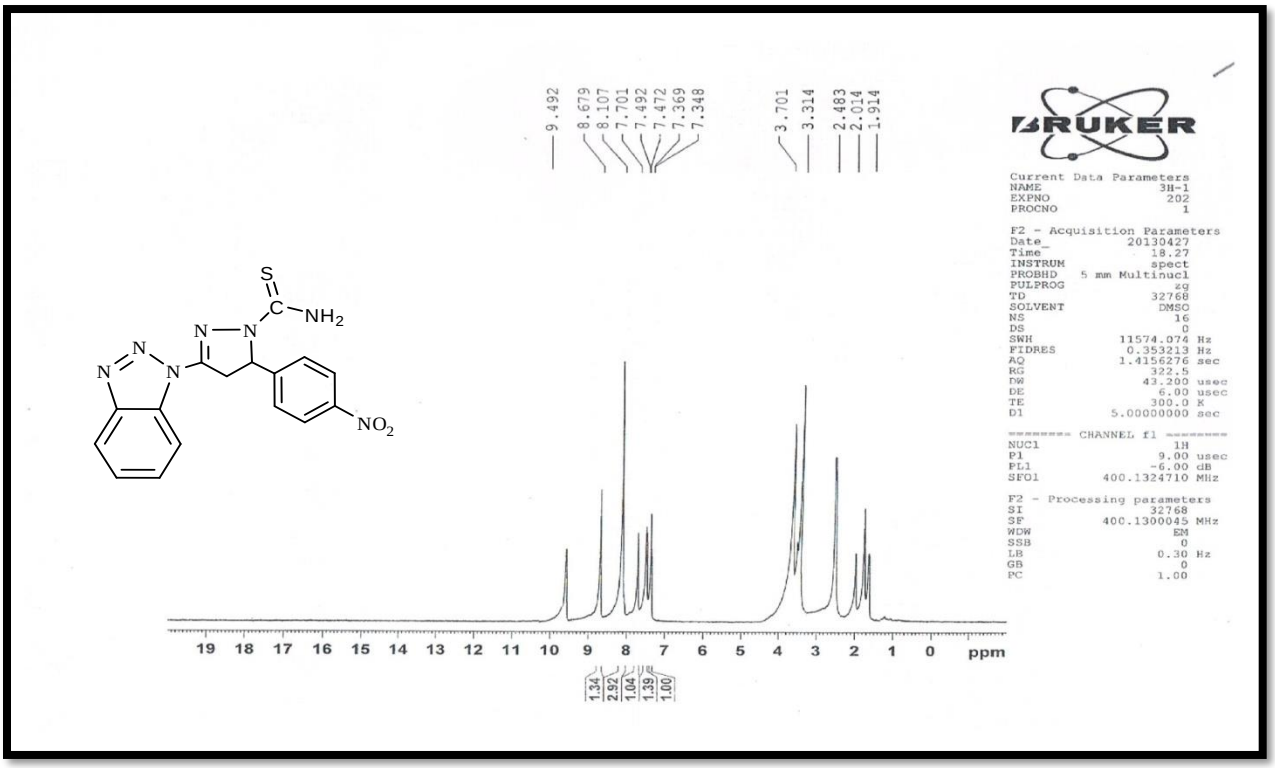
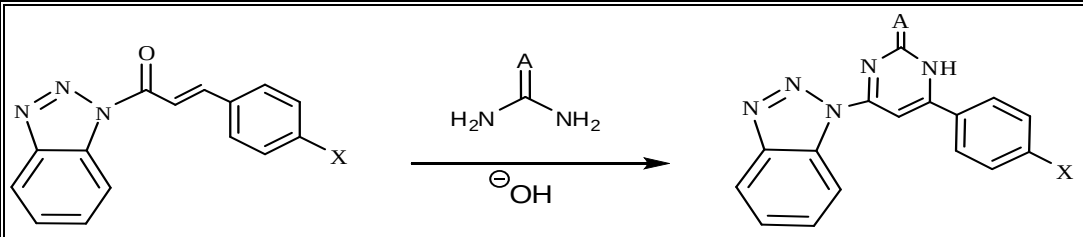


Figure [2] ^{1}H-NMR spectrum of the compound[C3].

Synthesis and identification of six heterocyclic membered ring . (pyrimidines).

The chalcones [A,B,C,D] was further reacted with urea, thiourea and guanidine in ethanol absolute to yield the corresponding pyrimidine derivatives by the following reaction .



X=N(CH$_3$)$_2$, OH, NO$_2$, Cl

A=O, S, N

Scheme[5]

The [C.H.N.S] analysis of synthesized compounds [A4,A5,A6,B4,B5,B6 ,C4,C5,C6,D4,D5,D6] was accepted agreement with the calculated percentage of elements were showed in table [5].

Table [5] C.H.N analysis, F.T.I.R data and the physical properties of pyremidine.

Com. No.	formula	Calculated			M. P ^{0}C	Yield %	R_f	*Infrared data (V,cm-1) (KBr disc)*
		C%	H%	N%				
A4	$C_{18}H_{16}N_6O$	(65.05) 64.49	(4.85) 4.55	(25.29) 25.01	114	90	0.6	3217(NH), 3049(C-Har),2952(C-Hal),1625(C=O),1600(C=C), 1546(C=N).
A5	$C_{18}H_{16}N_6S$				108	85	0.64	3253(NH), 3048(C-Har),2968(C-Hal), 1619(C=C), 1598(C=N).
A6	$C_{18}H_{17}N_7$	(65.24) 26.01	(5.17) 5.62	(29.59) 29.21	98	80	0.65	3404(NH), 3049(C-Har),2910(C-Hal), 1600(C=C), 1544(C=N).
B4	$C_{16}H_{11}N_5O_2$				102	85	0.74	3446(O-H),3346(NH),3080(C-Har),1625(C=O),1601(C=C), 1500(C=N).
B5	$C_{16}H_{11}N_5OS$	(59.80) 59.31	(3.45) 4.02	(21.79) 22.13	107	90	0.73	3277(O-H), 3277(NH), 3099(C-Har),1612(C=C),1573(C=N), 1307(C=S).
B6	$C_{16}H_{12}N_6O$				96	79	0.81	3440(O-H),3363(NH),3026(C-Har) , 1589(C=C), 1550(C=N).
C4	$C_{16}H_{10}N_6O_3$	(57.49) 57.12	(3.02) 2.53	(25.14) 24.61	105	75	0.73	3450(NH),3100(C-Har),1625 (C=O),1562(C=C), 1517(C=N),1371 (NO_2).
C5	$C_{16}H_{10}N_6O_2S$				110	80	0.8	3440(NH),3100(C-Har), 1600(C=C),1544(C=N), 1388(C=S),1348(NO_2).
C6	$C_{16}H_{11}N_7O_2$	(57.66) 57.24	(3.33) 3.01	(29.42) 29.87	92	78	0.66	3256(NH),3048(C-Har), 1614(C=C),1529(C=N), 1333(NO_2).
D4	$C_{16}H_{10}ClN_5O$				102	85	0.77	3217(NH),3032(C-Har), 1624(C=O), 1600(C=C). 1546(C=N),730(C-Cl).
D5	$C_{16}H_{10}ClN_5S$	(56.55) 56.01	(2.97) 2.54	(20.61) 20.13	116	80	0.65	3280(NH),3100(C-Har),1610 (C=C) , 1540(C=N), 1399 (C=S),730(C-Cl).
D6	$C_{16}H_{11}ClN_6$				95	75	0.73	3445(NH),3010(C-Har) 1586(C=C) ,1500(C=N),740(C-Cl).

The F.T.I.R spectra showed appearance of absorption band at (3445-3217) cm^{-1} of the symmetric stretching vibration of (-NH-) group and appearance stretching vibration bands between (1645-1624)$cm^{-1}$was due to the stretching vibration of (C=O) group of oxopyremidine cmpounds (A4,B4,C4 and D4), appearance stretching vibration bands between(1307-1390) due to (C=S) group of thiopyremidine (A5,B5,C5and D5) and disappearance stretching vibration bands between (1680-1660)$cm^{-1}$was due to the stretching vibration (C=O) of carbnyl group of all compounds. The remaining of the strong absorption band at 3446 cm^{-1} to the OH group in [B3] compound . All of these absorption bands are anther good evidence to formation this compounds.

^{1}H-NMR spectrum (δ ppm), Figure [3] of (A6) showed the following characteristic chemical signals (DMSO-*d*$_6$) as a solvent, ((6H), (N(CH$_3$)$_2$ 3.083), ((1H) (-CH-)$_{Pyremidine}$ 5.601), ((Ar-8H)) 7.348-8.083), ((-NH) 8.492).

^{1}H-NMR spectrum (δ ppm) of (B5) showed the following characteristic chemical signals (DMSO-*d*$_6$) as a solvent, ((1H), (-CH-) 5.331), ((Ar-8H)) 6.592-8.042), ((-NH) 8.682), ((OH) 9.539).

^{1}H-NMR spectrum (δ ppm) of (B6) showed the following characteristic chemical signals (DMSO-*d*$_6$) as a solvent, ((1H), (-CH-) 5.595), ((Ar-8H)) 6.851-7.588), ((-NH) 7.6), ((OH) 9.231).

^{1}H-NMR spectrum (δ ppm) of (C4) showed the following characteristic chemical signals (DMSO-*d*$_6$) as a solvent, ((1H), (-CH-) 5.312), ((Ar-8H)) 6.894-8.326), ((-NH) 8.501).

^{1}H-NMR spectrum (δ ppm) of (D4) showed the following characteristic chemical signals (DMSO-*d*$_6$) as a solvent, ((1H), (-CH-) 4.871), ((Ar-8H)) 6.973-7.969), ((-NH) 8.428).

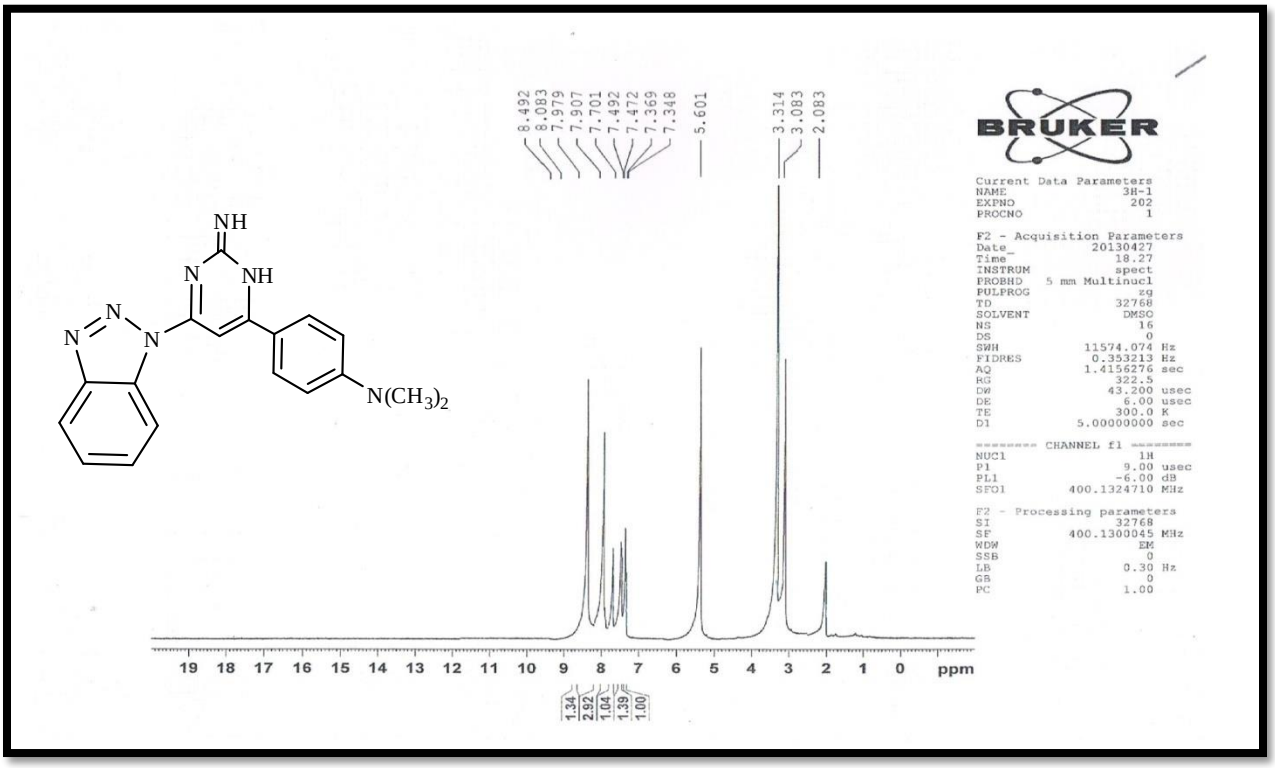
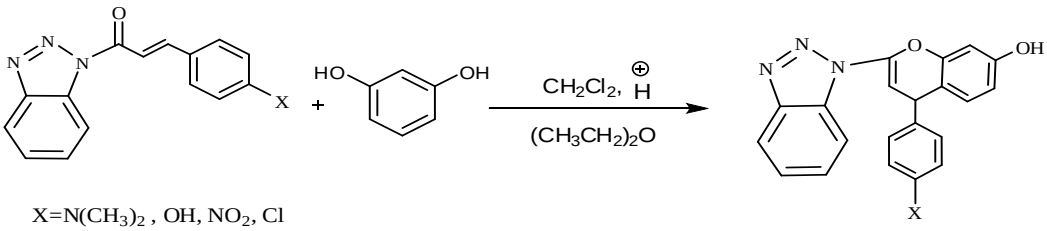


Figure [3] ^{1}H-NMR spectrum of the compound[A6].

5. Synthesis and identification of benzopyran.

The chalcones [A,B,C,D] was further reacted with resorcinol in diethylether ,dichlromethan and hydrochloric acid to yield the corresponding benzopyran derivatives by the following reaction.



Scheme[6]

The [C.H.N.S] analysis of synthesized compounds [A7 ,B7,C7,D7] was accepted agreement with the calculated percentage of elements were showed in table [6].

Table [6] C.H.N analysis, F.T.I.R data and The physical properties of benzopyran.

Com. No.	formula	Calculated			M. P ^{0}C	Yield %	R_f	*Infrared data (V,cm-1) (KBr disc)*
		C%	H%	N%				
A7	$C_{23}H_{20}N_4O_2$				140	79	0.74	3350(O-H),3053(C-Har) ,2955(C-Hal),1596(C=C), 1085(C-O-C).
B7	$C_{21}H_{15}N_3O_3$	(70.58) 70.32	(4.23) 3.82	(11.76) 11.23	125	85	0.64	3400(O-H),3002(C-Har), 1614(C=C), 1074(C-O-C).
C7	$C_{21}H_{14}N_4O_4$				160	80	0.6	3300(O-H),3008(C-Har), 1600(C=C),1380(NO_2), 1083(C-O-C).
D7	$C_{21}H_{14}ClN_3O_2$	(67.12) 66.32	(3.75) 3.15	(11.18) 11.05	135	75	0.63	3260(O-H),3002(C-Har), 1610(C=C),1091(C-O-C), 773(C-Cl).

The F.T.I.R spectra showed appearance strong absorption bands at (3400-3260) cm^{-1} of the symmetric stretching vibration of (OH) group, also appearance absorption bands at (1091-1074) cm^{-1} of the stretching vibration of (C-O-C) group and disappearance stretching vibration bands between (1680-1660)$cm^{-1}$was due to the stretching vibration of (C=O) group and disappearance stretching vibration bands between (1550-1600)$cm^{-1}$was due to the stretching vibration (C=C) of vinyl group of all compounds. All of these absorption bands are anther good evidence to formation [A7,B7,C7and D7].

^{1}H-NMR spectrum (δ ppm) of (A7) showed the following characteristic chemical signals (DMSO-d_6) as a solvent, ((6H), ($N(C\underline{H}_3)_2$ 3.033), ((-C=C<u>H</u>-C-) 4.548)$_{Pyran}$, ((Ar-H)) 6.399-7.872), ((OH) 9.428).

^{1}H-NMR spectrum (δ ppm), Figure [4] of (C7) showed the following characteristic chemical signals (DMSO-d_6) as a solvent, ((-C-C<u>H</u>-C) 4.797)$_{Pyran}$, ((Ar-<u>H</u>)) 6.607-8.701), ((OH) 9.869).

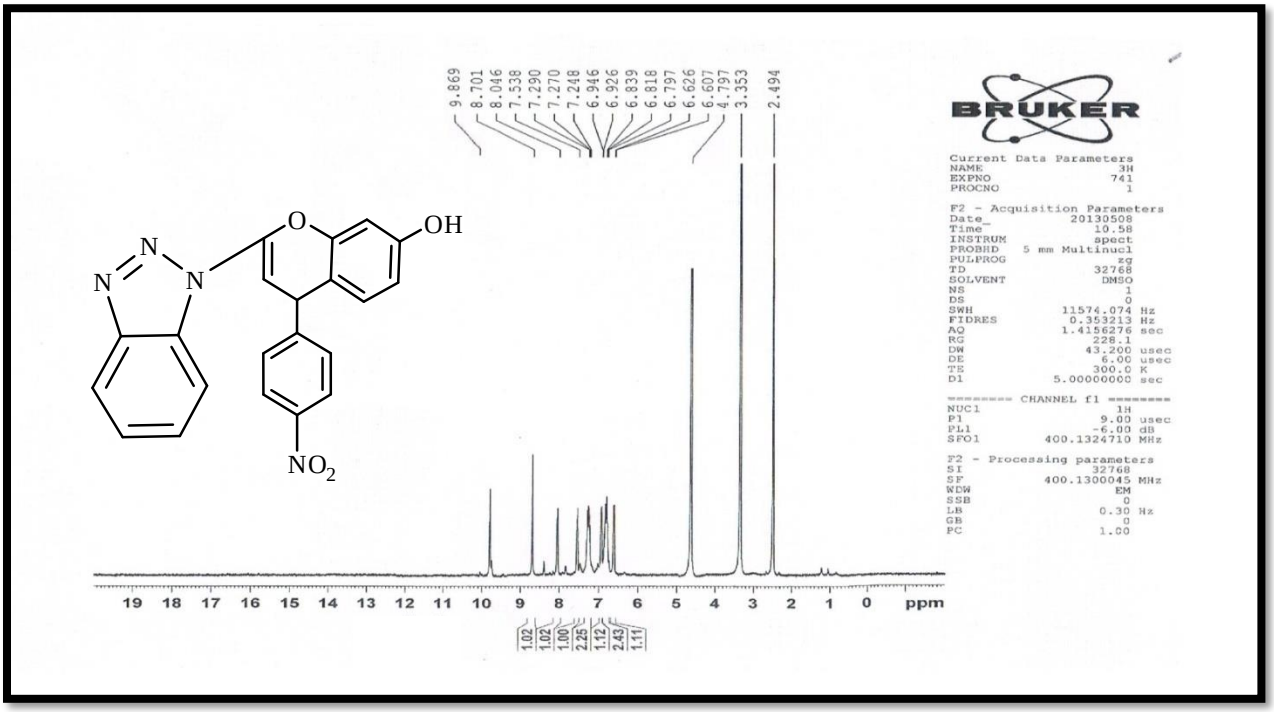
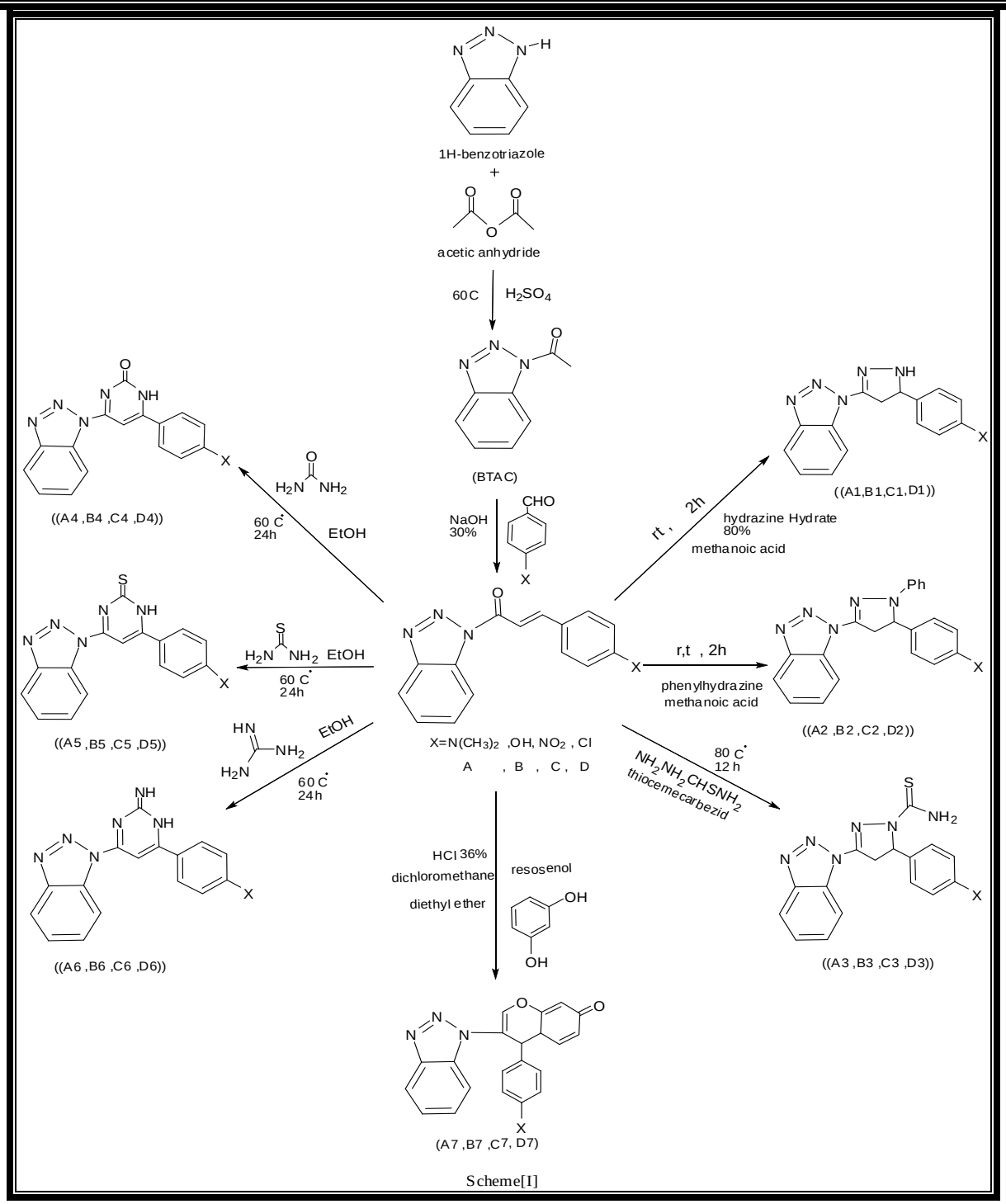


Figure [4] ^{1}H-NMR spectrum of the compound[C7].


1H-benzotriazole
+
acetic anhydride
60C
H_2SO_4
(BTAC)
NaOH 30%
CHO
X
60 C 24h
EtOH
((A4 ,B4 ,C4 ,D4))
r,t , 2h
hydrazine Hydrate 80%
methanoic acid
((A1,B1,C1,D1))
r,t , 2h
phenylhydrazine methanoic acid
((A2 ,B2 ,C2 ,D2))
EtOH
60 C 24h
((A5 ,B5 ,C5 ,D5))
EtOH
60 C 24h
((A6 ,B6 ,C6 ,D6))
80 C 12 h
$NH_2NH_2CHSNH_2$
thiocemecarbezid
((A3 ,B3 ,C3 ,D3))
X=N(CH3)2 ,OH, NO2 , Cl
A , B , C , D
HCl 36%
dichloromethane
diethyl ether
resosenol
(A7 ,B7 ,C7, D7)

Scheme[I]

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*تحضير وتشخيص ودراسة بعض الخواص الفيزياوية للمشتقات الجديده للبنزوتريازول

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الخلاصة

يتضمن البحث تحضير بعض المشتقات الغير متجانسة جديدة للبنزوتريازول مثل(البايروزولين ،البايرمدين و البزوبيران) التي لها اهميه كيميائيه و حياتيه.الخطوة الأولى تتضمن تحضير المركب 1-N استيل بنزوتريازول من تفاعل الاستك انهدريد مع البنزوتريازول بوجود حامض الكبريتيك .اما الخطوة الثانية تتضمن تحضير سلسلة من مركبات الكاربونيل الغير مشبعه(جالكونات) من تكاثف 1-N استيل بنزوتريازول مع الديهايدات الاروماتية المختلفه(4-(داي مثيل امنيو)بنزلديهايد ، 4-هيدروكسي بنزلديهايد ، 4- نيترو بنزلديهايد ،4- كلوروبنزلديهايد) بوجود القاعده تنتج مركبات جديده للجالكونات مثل

على التوالي) (A ,B ,C ,D

1 -3) N-(4-(داي مثيل امنيوفنيل) بروبينون) بنزوتريازول.

1-N (3-(4-(هيدروكسي فنيل) بروبينون) بنزوتريازول.

1 --3) N(4-(نيترو فنيل) بروبينون) بنزوتريازول.

1--3) N(4-(كلورو فنيل) بروبينون) بنزوتريازول.

لتحضير سلسلة من المركبات العضوية الغير متجانسه تعتبر الجالكونات المحضره في الخطوه السابقه الممهد لتحضير عدد من هذه المركبات الحلقيه من تفاعلها مع :

1- الهيدرازين ،الفنيل هيدرازين في الوسط حامضي (حامض ميثانويك) تنتج سلسله من مركبات

البايروزولين (A1,A2, B1 ,B2, C1 ,C2, D1, D2).

2-الثايو سيميكربزايد لتكوين مشتقات اخرى للبايروزولين (A3 ,B3 ,C3 ,D3)

3-اليوريا ،والثايويوريا والكواندين لتكوين مشتقات البايريميدين

(A4,A5,A6 ,B4 ,B5 ,B6, C4, C5 ,C6,D4 ,D5, D6).

4-الرزوسينول في الوسط الحامضي (حامض الهيدروكلوريك)لتكوين مشتقات جديده للبنزوبايران

(A7 ,B7 ,C7 ,D7).

***البحث مستل من رسالة ماجستير للباحث الأول .**