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Research article

Rapid And Convenient Microwave-assisted, Regioselective Synthesis and Characterization of a Novel 2,5-Bis(heteroamino)-1,4-Benzoquinones

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Abstract

A series of twenty four 2, 5-Bis(aminoheterocycles) -1,4-benzoquinones were prepared in yields ranging from 95-74% via a reaction between hydroquinone and various heteroamine moiety,in methanolic solution under microwave irradiation,the last was synthesized in three main steps;firstly chalcones produced by the claisen Schmidt condensation of aminoacetophenone with differentpara substitutedbenzyldehydes in ethanolic KOH solution under microwave irradiation.These chalcones werefurther reacted with (semicarbazide,thiosemicarbazide,urea and thiourea), respectively in the presence of base in ethanol,resultedpyrazole and pyrimidine derivatives. Michael 1,4- nucleophlic addition tookplace between amino heterocycles derivatives and hydroquinone in one pot aerobic oxidationunder microwave irradiation resulting in a novel2,5Bis(pyrazoleamino)-1,4-benzoquinones and 2,5 Bis (pyrimimdineamino)-1,4-benzoquinones .The newlysynthesized aminoquinones derivatives were characterized on the basis of their chemical propertiesand spectroscopic(^{13}C NMR,^{1}H NMR,FT IR,CHN), all analysis data showed full agreement with the suggested structures.

Keywords: RegioselectiveSynthesis,Aminoquinones , Heterocyclic , Microwave teqnique

Introduction

Quinones are ubiquitous in nature (1),they are tremendous group of naturally occurring pigments (2). They have captivated human concern for thousands of year, at first in due to their bright colors with possible uses as dyes and drugs. Pigments of diverse colors isolated from different provenances,have been depicted as quinonid compounds. (3, 4)

These class of compounds are dispersed vastly innature (5,6) they play pivotal role in biological functions, inclusive biological redox process,their role as electron transfer agents in primary metabolic process such as

photosynthesis and respiration is vital to human.(7) Enormous of benzoquinone derivatives displays eminent pharmacological applications(8) such as antibiotic(9), anticoagulant, antineoplastic antimalarial cancer chemotherapy, for example doxorubicin which has aquinonedmoeity being one of the front line cancer chemotherapy treatments in the UK(10-12).

Quinone analogs create great interest among naturally occurring compounds (13), they are habitual specimen that posses versatile pharmacological activity ranging from antitumor, inhibition of the HIV 1 reverse transcriptase to antibacterial and antiprtozols.(14) The regioselectivity of nucleophlic addition to 1,4-benzoquinones were tremendously studied by means of theory and experiment, results of these studies illustrated the nucleophlicnature that would mainly attack the 5-position of donor substituted 1,4-benzoquinone and 3-position of acceptor –substituted 1,4-benzoquinone.when the nucleophile is nitrogen atom version of the Michael addition is often referred to as the aza-Michael reaction, the inclusion of a nitrogen functionality within, quinones Furnishes to unique systems, as in aminoquinone derivatives, enhances their versatility as building blocks for the construction of biologically (15) compound libraries and qualifies them as potential precursors.itsreported the conjugate addition of amines to carbon–carbon double bonds is a useful protocol in synthetic organic chemistry. It is used extensively in the synthesis of pharmaceutical intermediates, peptide analogues, antibiotics, and other biologically active molecules and drugs (16).The nucleophlic addition of amine to 1,4-benzoquinone scaffold afford novel protocol to synthesis aminoquinone analogues with high regioselectivty..Microwave-assisted chemistry displays modernistic possibilities for the advancement of any chemical reaction that is thermally possible. It typically leads up to prompter reactions, elevates yields and diminishes the formation of by-products.

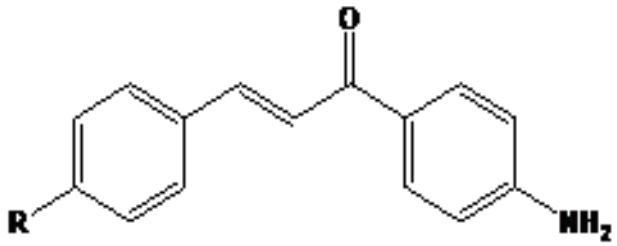
The prominence of these valuable aminoquinones compounds stimulate interest in conducting the present study which was aiming at synthesizing a new analogues of 2,5-diaminoquinones incorporated with two bioactive moiety : quinones unit and pyrazole, using microwave technique.

Synthesis of new compounds:

The synthesis of 2,5-diaminoquinones derivatives consists of three main steps:

i)Synthesis of Chalcones

A mixture of 0.01 mole of p-aminoacetophenone, 0.01mole of substituted benzyldehyde were dissolved in 3ml of ethanol and 0.112gm KOH then irradiated inside microwave oven 115W for 4min. and then it was poured in to crushed ice and acidified with HCL .The solid separated was filtered and recrystallized from ethanol. The physical properties were illustrated in table (1), chalconecan be represented by the following structure.

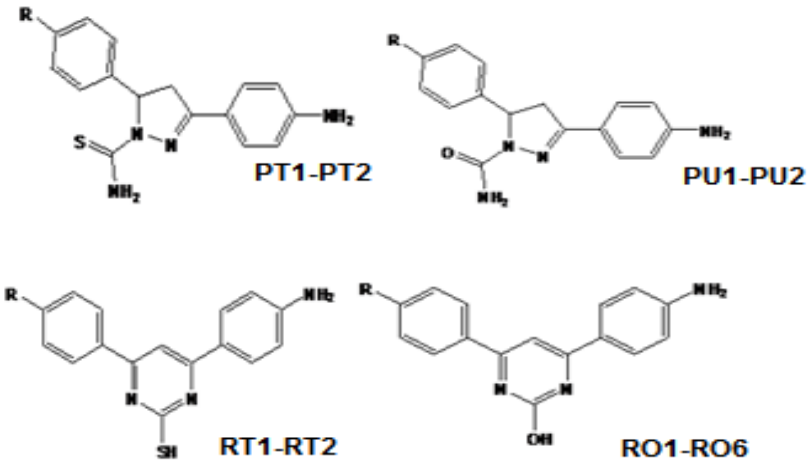


Comp. Code	R	Mol Formula	Mol.Wt (gm)	%Yield	M.P c°	Reaction Time (Min) / energy(W)	R_f	appearance
CH-1	-F	$C_{15}H_{12}FNO$	241.09	95	152-154	2.5 / 400	0.64	Yellow/powder
CH-2	-Cl	$C_{15}H_{12}ClNO$	257.06	88	182-183	2.9 / 400	0.72	Yellow/powder
CH-3	-Br	$C_{15}H_{12}BrNO$	302.17	86	212-214	3.5 / 450	0.77	yellow/powder
CH-4	$-NO_2$	$C_{15}H_{12}N_2O_3$	268.08	96	182-184	4 / 450	0.66	Red orange /powder
CH-5	-OH	$C_{15}H_{13}NO_2$	239.09	84	251-253	6 /500	0.55	maroon /powder
CH-6	$-CH_3$	$C_{16}H_{15}NO$	237.12	79	163-165	6 /500	0.81	cream /powder

Table(1): physical data, percentage of yields, energy and reaction time for chalcones

ii)Synthesis of(pyrozole,pyrimidine)derivatives:

A mixture of (chalcone) (0.01 mole) (semicarbazide,thiosemicarbazideurea,thiourea)respectively (0.02 mole) and KOH (0.02 mole; 1.12 g) in 10 ml ethanol. The contents were thoroughly mixed. The reaction mixture under goes to microwave irradiation in a commercially available IFB domestic microwave oven having a maximum power output of 480W operating at 2450Hz intermittently at 30 seconds intervals for 3-6 min on a completion of reaction as monitored by TLC. It was then cooled and poured in cold water acidified with dil.HCl. Filtered, washed and dried. The product was recrystallized from ethanol to get product. The purity of the compound was checkedwith TLC. The physical properties of the synthesized compounds were illustrated in tables (2-5), these compounds can be represented by the following structures:



Table(2): physical data ,percentage of yields, energy and reaction time for pyrazole urea

Comp. Code	R	Mol Formula	Mol.Wt (gm)	%Yield	M.P c°	ReactionTime (Min) / energy(W)	R_F	appearance
PU-1	-F	**C16H15FN4O**	**298.12**	**92**	**226-228**	3/214	0.59	**Browne reddish/powder**
PU-2	-Cl	**C16H15ClN4O**	**314.09**	**88**	**235-237**	3/214	0.88	**Browen/powder**
PU-3	-Br	**C16H15BrN4O**	**359.22**	**86**	**234-236**	3/214	0.95	dark golden rod/powder
PU-4	$-NO_2$	**C16H15N5O3**	**325.32**	**93**	**336-337**	3/200	0.59	dark orange/powder
PU-5	-OH	**C16H16N4O2**	**296.32**	**84**	**204-206**	3/315	0.61	Firebrick/powder
PU-6	$-CH_3$	**C12H18N4O**	**294.15**	**77**	**239-241**	3/310	0.77	Crimson/powder

Table (3): physical data, percentage of yields, energy and reaction time for pyrazolethiourea

Comp. Code	R	Mol Formula	Mol.Wt (gm)	%Yield	M.P c°	ReactionTime (Min) / energy(W)	R_F	appearance
PT-1	-F	**$C_{16}H_{15}FN_4S$**	**314.10**	**92**	**263-264**	3/214	0.83	**Light orange/powder**
PT-2	-Cl	**$C_{16}H_{15}ClN_4S$**	**330.07**	**88**	**371-372**	3/214	0.75	**persimmon/powder**
PT-3	-Br	**$C_{16}H_{15}BrN_4S$**	**375.29**	**86**	**277-279**	3/214	0.78	Light yellow/powder
PT-4	$-NO_2$	**$C_{16}H_{15}N_5O_2S$**	**341.39**	**93**	**286-288**	3/200	0.61	Orange red/powder

PT-5	-OH	**$C_{16}H_{16}N_4OS$**	**312.39**	**84**	**290-291**	3/315	0.53	garnet/powder
PT-6	-CH_3	**$C_{17}H_{18}N_4S$**	**310.42**	**77**	**273-275**	3/310	0.99	Satin gold/powder

Table(4): physical data ,percentage of yields, energy and reaction time for pyrimidine -ol

Comp. Code	**R**	**Mol Formula**	**Mol.Wt (gm)**	**%Yield**	**M.P c°**	**ReactionTime (Min) / energy(W)**	**R_F**	**appearance**
RO-1	-F	**$C_{16}H_{12}FN_3O$**	**281.10**	**92**	**213-214**	3/214	0.72	**Light orange/powder**
RO-2	-Cl	**$C_{16}H_{12}ClN_3O$**	**297.07**	**88**	**218-220**	3/214	0.84	**persimmon/powder**
RO-3	-Br	**$C_{16}H_{12}BrN_3O$**	**342.19**	**86**	**177-179**	3/214	0.77	Light yellow/powder
RO-4	-NO_2	**$C_{16}H_{12}N_4O_3$**	**308.29**	**93**	**217-219**	3/200	0.68	Orange red/powder
RO-5	-OH	**$C_{16}H_{13}N_3O_2$**	**279.29**	**84**	**187-188**	3/315	0.56	garnet/powder
RO-6	-CH_3	**$C_{17}H_{15}N_3O$**	**277.32**	**77**	**222-224**	3/310	0.92	Satin gold/powder

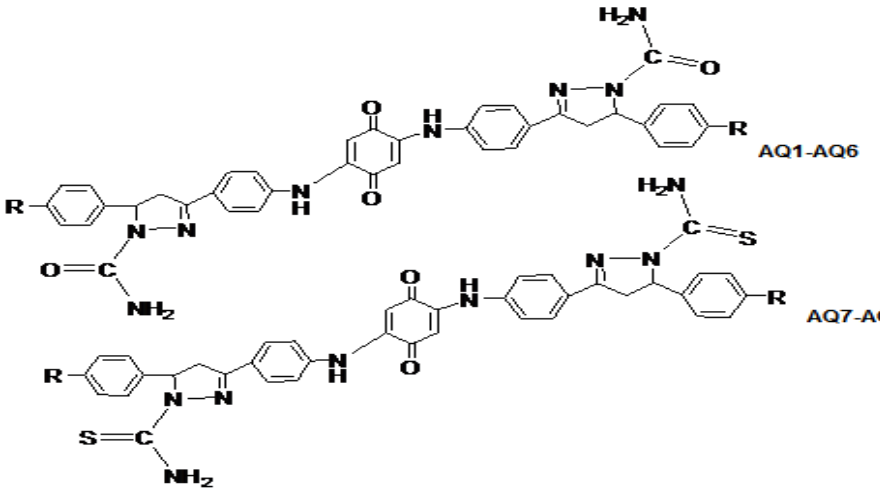
Table(5): physical data ,percentage of yields, energy and reaction time for pyrimidine -thyol

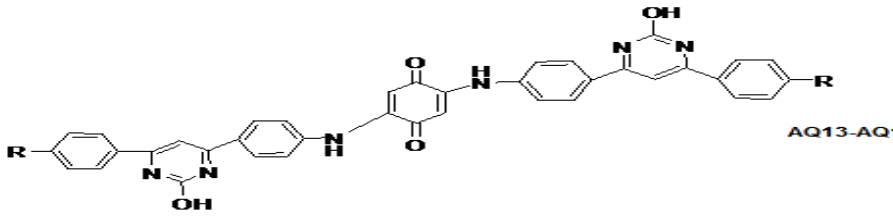
Comp. Code	**R**	**Mol Formula**	**Mol.Wt (gm)**	**%Yield**	**M.P c°**	**ReactionTime (Min) / energy(W)**	**R_F**	**appearance**
RT-1	-F	**$C_{16}H_{12}FN_3S$**	**279.07**	**92**	**273-274**	3/214	0.62	**Light orange/powder**

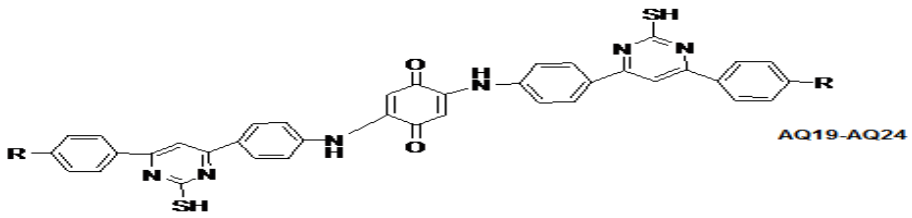
RT-2	-Cl	**$C_{16}H_{12}ClN_3S$**	**313.80**	**88**	**286-287**	3/214	0.73	**persimmon/powder**
RT-3	-Br	**$C_{16}H_{12}BrN_3S$**	**358.26**	**86**	**188-190**	3/214	0.78	Light yellow/powder
RT-4	-NO_2	**$C_{16}H_{12}N_4O_2S$**	**324.36**	**93**	**298-299**	3/200	0.82	Orange red/powder
RT-5	-OH	**$C_{16}H_{13}N_3OS$**	**295.08**	**84**	**101-103**	3/315	0.71	garnet/powder
RT-6	-CH_3	**$C_{17}H_{15}N_3S$**	**293.39**	**77**	**227-228**	3/310	0.89	Satin gold/powder

iii)Synthesis of 2,5-bis amino-p-benzoquinone derivatives:

Methanol solution (10ml) of amine (hetrocyclicseries)(2.3 mmole) was added to methanol solution (10 ml) of hydroquinones (0.05 mmole).This mixture was irradiated at (320w) for two minutes .the solution was evaporated in room temperature then product was purified by recrystalization from methanol toobtain long crystals with different colors (red,orange, yellow), physical properties were displayed in table(1-6). the structure of final products can be represented by following structures:







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Table(6): physical data ,percentage of yields, energy and reaction time for 2,5-diaminoquinone

Comp. Code	R	Mol Formula	Mol.Wt (gm)	%Yield	M.P c°	Reaction Time (Min) / energy(W)	R_f	appearance
AQ-1	-F	**$C_{38}H_{30}F_2N_8O_4$**	**700.69**	78	152-154	3/214	0.72	**Light orange/crystal**
AQ-2	-Cl	**$C_{38}H_{30}Cl_2N_8O_4$**	**733.69**	83	162-164	3/214	0.61	**Dark yellow/crystal**
AQ-3	-Br	**$C_{38}H_{30}Br_2N_8O_4$**	**822.50**	80	130-132	3/214	0.63	Light yellow/powder
AQ-4	-NO_2	**$C_{38}H_{30}N_{10}O_8$**	**754.22**	92	148-150	3/200	0.54	Orange red/powder
AQ-5	-OH	**$C_{38}H_{32}N_8O_6$**	**696.71**	86	169-170	3/315	0.81	garnet/powder
AQ-6	-CH_3	**$C_{40}H_{36}N_8O_4$**	**692.29**	84	125-124	3/310	0.64	Crimson /powder
AQ-7	-F	**$C_{38}H_{30}F_2N_8O_2S_2$**	**732.19**	74	122-123	3/214	0.86	Gold/crystal
AQ-8	-Cl	**$C_{38}H_{30}Cl_2N_8O_2S_2$**	**765.73**	89	134-136	3/214	0.68	Light yellow/crystal
AQ-9	-Br	**$C_{38}H_{30}Br_2N_8O_2S_2$**	**854.64**	94	186-188	3/214	0.7	Reddish Browne/crystal
AQ-10	-NO_2	**$C_{38}H_{30}N_{10}O_6S_2$**	**786.84**	94	187-189	3/214	0.49	Red orange/long crystal
AQ-11	-OH	**$C_{38}H_{32}N_8O_4S_2$**	**728.84**	91	144-146	3/200	0.41	Firebrick/crystal
AQ-12	-CH_3	**$C_{40}H_{36}N_8O_2S_2$**	**724.90**	90	135-136	3/214	0.62	Crimson/crystal
AQ-13	-F	**$C_{38}H_{24}F_2N_6O_4$**	**666.18**	88	176-177	3/200	0.81	Bright yellow/crystal
AQ-14	-Cl	**$C_{38}H_{24}Cl_2N_6O_4$**	**699.54**	91	128-130	3/214	0.92	Yellow/crystal
AQ-15	-Br	**$C_{38}H_{24}Br_2N_6O_4$**	**788.44**	86	97-99	3/310	0.97	Browne/ crystal
AQ-16	-NO_2	**$C_{38}H_{24}F_2N_8O_8$**	**720.17**	81	176-177	3/200	0.85	Dark orange /crystal
AQ-17	-OH	**$C_{38}H_{26}N_6O_6$**	**662.65**	91	98-100	3/310	0.79	Reddish brown/ long crystal
AQ-18	-CH_3	**$C_{40}H_{30}N_6O_4$**	**658.70**	85	142-144	3/200	0.66	Cream/crystal
AQ-19	-F	**$C_{38}H_{24}F_2N_6O_2S_2$**	**698.76**	93	111-113	3/214	0.74	Yellow/crystal
AQ-20	-Cl	**$C_{38}H_{24}Cl_2N_6O_2S_2$**	**731.67**	88	148-151	3/200	0.68	Light yellow /crystal

AQ-21	-Br	**$C_{38}H_{24}Br_2N_6O_2S_2$**	**820.57**	79	187-189	3/310	0.69	Bright yellow/long crystal
AQ-22	-NO_2	**$C_{38}H_{24}N_8O_6S_2$**	**752.78**	92	173-175	3/214	0.56	Light orange /long crystal
AQ-23	-OH	**$C_{38}H_{26}N_6O_4S_2$**	**694.15**	95	129-131	3/310	0.41	Maroon/long crystal
AQ-24	-CH_3	**$C_{40}H_{30}N_6O_2S_2$**	**690.84**	92	164-166	3/214	0.93	Red orange /long crystal

Materials and methods:

Melting points were determined in open capillary tubes and were found uncorrected. The synthesized compounds are characterized and identified by elemental analysis, IR spectra were recorded on FT-IR spectrometer (Perkin Elmer) using KBr disc method, ^{13}C spectra were recorded on(Bruker MHz) and 1HNMR spectra were recorded on 1H FT-NMR (Bruker AMX 400 MHz) spectrometer in $CDCl_3$. the FT-IR and melting point measurement were carried out in Iraq ,Basrah university ,science college ,chemistry department .the^{13}CNMR, ^{1}HNMR and CHN measurement were carried in Iran, Tehran , TarbiatModares University.

Results and Discussion

3,3'-(4,4'-(3,6-dioxocyclohexa-1,4-diene-1,4-diyl)bis(azanediyl)bis(4,1-phenylene))bis(5-(4-fluorophenyl)-4,5-dihydro-1H-pyrazole-1-carboxamide) (AQ1) : Yeild: (78%),mp(152-154) .IR (KBr pellet, cm $^{-1}$):U(C=Oquinone)1636.59, U(C=Oamide) 1650.90, U(C-N)1284.50, U(C=N)1594.3, U(C= C)1600.81, U(C-Haromatic) 30.58.89, U(C-Haliphatic) 2978.53-2891.10. U(NH2asym, sym) 3481.70,3328.98. U(C-F) 1220.86 ;^{1} H NMR δ($CDCl_3$): 4.23(S,4H –NH2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S, 2H –NH); 6.722-6.700[d, J = 8.8 Hz,4 H –C(3,3',3",3"')] ;7.647-7.626 [d,J =8.4Hz,4 H –C(4,4',4",4"')];7.261-7.230 [d, J 12.4=Hz,4 H –C(5,5',5",5")]; 7.331-7.300 [d, J =12.4 Hz,4 H –C(6,6',6",6")]; 3.752-3.717 [m, J=14 Hz 4H –C(7,7',8,8')];4.121-4.086 [m, J=14 Hz, 2H –C(9,9')];^{13} C- NMR δ($CDCl_3$) :181.03[C(1), C(1')]; 105.39 [C(2), C(2')]; 152.31[C(3), C(3')]; 142.98[C(4), C(4')] ; 116.90-116.88 [C(5), C(5')]; 130.74-130.70[C(6), C(6')]; 129.60 [C(7), C(7')]; 152.71[C(8), C(8')]; 39.20[C(9), C(9')]; 61.09[C(10), C(10')]; 155.32 [C(11), C(11')]; 140.88[C(12), C(12')];129.28-129.20[C(13), C(13')]; 115.75-115.73[C(14), C(14')]; 160.25[C(15), C(15')];Anal.(%)for $C_{38}H_{30}F_2N_8O_4$, Calcd.C, 65.14;H, 4.32;N, 15.99;Found C, 65.16; H,4.30;N,15.90.

3,3'-(4,4'-(3,6-dioxocyclohexa-1,4-diene-1,4-diyl)bis(azanediyl)bis(4,1-phenylene))bis(5-(4-chlorophenyl)-4,5-dihydro-1H-pyrazole-1-carboxamide)(AQ2) : Yield : (83%), mp.164-162, IR (KBr pellet, cm^{-1}):U(C=Oquinone)1635.56, U(C=Oamide) 1650.91, U(C-N)1244.28, U(C=N)1591.33, U(C= C)1601.58, U(C-Haromatic) 3051.80, U(C- Haliphatic) 2987.28-2811.19. U(NH2asym, sym) 3481.73,3388.94. U(C-Cl) 1100.91. ; 1 H NMR δ: 4.23(S,4H –NH2), 6.306-6.6332(S,2H –C(1,1'), 2.301(br. S, 2H –NH), 6.722-6.700[d, J =8.8 Hz,4 H –C(3,3',3",3"')],7.647-7.626 [d, J = 8.4 Hz,4 H –C(4,4',4",4"')],7.311-7.299 [d, J =4.8 Hz,4 H –C(5,5',5",5")],7.201-7.191[d, J =4.0Hz,4 H –C(6,6',6",6")], 3.752-3.709 [m, J =14Hz, 4H –C(7,7',8,8')],4.121-4.086 [m, J =14Hz,2H –C(9,9')];^{13} C- NMR δ($CDCl_3$) :181.03[C(1), C(1')] ; 105.39 [C(2), C(2')]; 152.31[C(3), C(3')]; 142.98[C(4), C(4')] ; 116.90-116.88 [C(5), C(5')]; 130.74-130.70[C(6), C(6')]; 129.60 [C(7), C(7')]; 152.71[C(8), C(8')]; 39.21 [C(9), C(9')]; 60.09 [C(10), C(10')]; 155.32 [C(11), C(11')]; 144.98 [C(12), C(12')]; 127.11-127.10 [C(13), C(13')]; 128.02-128.00 [C(14), C(14')]; 132.26 [C(15), C(15')] ;Anal.(%)for $C_{38}H_{30}Cl_2N_8O_4$, Calcd.C: 62.21,H: 4.12,N: 15.27;Found:C : 62.22, H: 4.19, N:15.24.

3,3'-(4,4'-(3,6-dioxocyclohexa-1,4-diene-1,4-diyl)bis(azanediyl)bis(4,1-phenylene))bis(5-(4-bromophenyl)-4,5-dihydro-1H-pyrazole-1-carboxamide)(AQ3): Yield : (80%), mp.132-130 .IR (KBr pellet, cm $^{-1}$):U(C=Oquinone) 1631.59, U(C=Oamide) 1650.90, U(C-N) 1284.50, U(C=N) 1589.73, (C= C) 1600.56, U(C- Haromatic) 3055.62, U(C- Haliphatic2987.13-2881.59 .U(NH2asym, sym) 3481.70, 3328.O6. U(C-Cl) 1091.12;^{1} H NMR($CDCl_3$) δ: 4.23(S,4H –NH2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S, 2H –NH); 6.722-6.700[d, J = Hz,4 H –C(3,3',3",3"')] ;7.647-7.626 [d, J = 8.8 Hz,4 H –C(4,4',4",4"')];7.282-7.243 [d, J

=15.6Hz,4 H –C(5,5',5",5")]; 7.999-7.961[d, J =15.2 Hz,4 H –C(6,6',6",6")]; 3.752-3.717 [m, J =14Hz, 4H –C(7,7',8,8')];4.121-4.086 [m, J =14Hz, 2H –C(9,9')];[13] C- NMR($CDCl_3$) δ :181.03[C(1), C(1')] ; 105.39 [C(2), C(2')]; 152.31[C(3), C(3')]; 142.98[C(4), C(4')] ; 116.90-116.88 [C(5), C(5')]; 130.74-130.70[C(6), C(6')]; 129.60 [C(7), C(7')]; 152.71[C(8), C(8')]; 39.20 [C(9), C(9')]; 61.09 [C(10), C(10')]; 155.32 [C(11), C(11')]; 145.38 [C(12), C(12')]; 127.33-127.30 [C(13), C(13')]; 131.67-131.66 [C(14), C(14')]; 121.95 [C(15), C(15')];Anal.(%) for $C_{38}H_{30}Br_2N_8O_4$Calcd. C: 55.49, H: 3.68,N: 13.62,Found: C: 55.56, H: 3.71,N: 13.60.

3,3'-(4,4'-(3,6-dioxocyclohexa-1,4-diene-1,4-diyl)bis(azanediyl)bis(4,1-phenylene))bis(5-(4-nitrophenyl)-4,5-dihydro-1H-pyrazole-1-carboxamide) (AQ4). Yield 92%,mp.148-150,IR (KBr pellet, cm $^{-1}$):U(C=Oquinone) 1639.52, U(C=Oamide) 1650.93, U(C-N) 1284.86, U(C=N) 1557.22, U(C= C) 1600.52, U(C- Haromatic) 3052.73, U(C- Haliphatic)2982.16-2886.67. U(NH2asym, sym) 3483.79, 3328.28;[1] H NMR($CDCl_3$) δ: 4.23(S,4H –NH2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S, 2H –NH);6.695-6.674[d, J =8.4 Hz,4 H –C(3,3',3",3'")] ;7.647-7.626 [d, J =8.4Hz,4 H –C(4,4',4",4'")];7.454-7.415 [d, J =15.6 Hz,4 H –C(5,5',5",5")]; 8.339-8.300[d, J =15.6Hz,4 H –C(6,6',6",6")]; 3.752-3.717 [m, J =14Hz 4H –C(7,7',8,8')];4.121-4.086 [m, J =14Hz 2H –C(9,9')];[13] C- NMR ($CDCl_3$) δ :181.03[C(1), C(1')] ; 105.39 [C(2), C(2')]; 152.31[C(3), C(3')]; 142.98[C(4), C(4')] ; 116.90-116.88 [C(5), C(5')]; 130.74-130.70[C(6), C(6')]; 129.60 [C(7), C(7')]; 152.71[C(8), C(8')]; 39.20 [C(9), C(9')]; 61.09 [C(10), C(10')]; 155.32 [C(11), C(11')]; 147.32 [C(12), C(12')]; 123.63-123.60 [C(13), C(13')]; 123.76-123.77 [C(14), C(14')]; 145.9 [C(15), C(15')];Anal.(%) for $C_{38}H_{30}N_{10}O_8$, Calcd.C:60.47,H:4.01,N: 18.56;Found: . C:60.37, H: 4.03, N: 18.86.

3,3'-(4,4'-(3,6-dioxocyclohexa-1,4-diene-1,4-diyl)bis(azanediyl)bis(4,1-phenylene))bis(5-p-tolyl-4,5-dihydro-1H-pyrazole-1-carboxamide)(AQ5). Yeild86%, mp.169-171, IR(KBr)pellet, cm^{-1}):U(C=Oquinone) 1627.81, U(C=Oamide) 1658.61, U(C-N 1238.21, U(C=N) 1569.92, U(C= C) 1595.02, U(C- Haromatic) 3026.11, U(C- Haliphatic) 2910.30-2852.57, U(NH2asym,sym) 3490.93, 3346.24;[1] H NMR($CDCl_3$) δ: 4.23(S,4H –NH2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S, 2H –NH);6.811-6.814[d, J =1.2 Hz,4 H –C(3,3',3",3'")] ;7.875-7.873 [d, J =0.8 Hz,4 H –C(4,4',4",4'")];7.151-7.147 [d, J =1.6Hz,4 H –C(5,5',5",5")]; 6.933-6.929[d, J =1.6 Hz,4 H –C(6,6',6",6")]; 3.752-3.717 [m, J =14 Hz, 4H –C(7,7',8,8')];4.121-4.086 [m, J =14 Hz, 2H –C(9,9')],2.51[S 6H-CH_3)];[13] C- NMR ($CDCl_3$) δ :181.03[C(1), C(1')] ; 105.39 [C(2), C(2')]; 152.31[C(3), C(3')]; 142.98[C(4), C(4')] ; 116.90-116.88 [C(5), C(5')]; 130.74-130.70[C(6), C(6')]; 129.60 [C(7), C(7')]; 152.71[C(8), C(8')]; 39.20 [C(9), C(9')]; 61.09 [C(10), C(10')]; 155.32 [C(11), C(11')]; 137.42 [C(12), C(12')]; 125.03-125.00 [C(13), C(13')]; 128.77-128.73 [C(14), C(14')]; 138.05 [C(15), C(15')]; 21.65 [C(16), C(16')];Anal.% for $C_{40}H_{36}N_8O_4$Calcd.C: 69.34,H: 5.13,N: 16.11; Found: C: 69.35,H: 5.24,N: 16.17.

3,3'-(4,4'-(3,6-dioxocyclohexa-1,4-diene-1,4-diyl)bis(azanediyl)bis(4,1-phenylene))bis(5-(4-hydroxyphenyl)-4,5-dihydro-1H-pyrazole-1-carboxamide) (AQ6). Yeild84%, mp.124-125;IR (KBr pellet, cm $^{-1}$):U(C=Oquinone) 1631.13, U(C=Oamide) 1659.11, U(C-N) 1288.36, U(C=N) 1571.88, U(C= C) 1595.02, U(C- Haromatic) 3072.24, U(C- Haliphatic) 2910.30-2852.57. U(NH2asym, sym) 3490.92, 3355.91, U(O- H) 3222.83;[1] H NMR ($CDCl_3$)δ: 4.23(S,4H –NH2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S, 2H –NH);6.722-6.700[d, J =8.8Hz,4 H –C(3,3',3",3'")] ;7.647-7.626 [d, J = 8.4 Hz,4 H –C(4,4',4",4'")];7.261-7.241 [d, J =8.00 Hz,4 H –C(5,5',5",5")]; 7.321-7.2999[d, J =8.8 Hz,4 H –C(6,6',6",6")]; 3.752-3.717[m, J =14 Hz, 4H –C(7,7',8,8')];4.121-4.086 [m, J =14 Hz, 2H –C(9,9')]; 9.472[S,2H-C(11,11')];[13] C- NMR ($CDCl_3$) δ :181.03[C(1), C(1')] ; 105.39 [C(2), C(2')]; 152.31[C(3), C(3')]; 142.98[C(4), C(4')] ; 116.90-116.88 [C(5), C(5')]; 130.74-130.70[C(6), C(6')]; 129.60 [C(7), C(7')]; 152.71[C(8), C(8')]; 39.20 [C(9), C(9')]; 61.09 [C(10), C(10')]; 155.32 [C(11), C(11')]; 135.24 [C(12), C(12')]; 127.44127.42 [C(13), C(13')]; 115.29-115.27 [C(14), C(14')]; 156.5 [C(15), C(15'); Anal.% for $C_{38}H_{32}N_8O_6$Calcd .C: 65.51, H: 4.63,N: 16.08, Found:C,65.56; H, 4.51; N, 15.99.

3,3'-(4,4'-(3,6-dioxocyclohexa-1,4-diene-1,4-diyl)bis(azanediyl)bis(4,1-phenylene))bis(5-(4-fluorophenyl)-4,5-dihydro-1H-pyrazole-1-carbothioamide) (AQ7): Yeild74%, mp.122-123, IR (KBr pellet, cm $^{-1}$):U(C=Oquinone) 1636.13, U(S=O) 1286.43, U(C=N) 1589.23, U(C= C) 1602.74, U(C- Haromatic) 3058.80, U(C- Haliphatic) 2987.53-2891.11 U(NH2asym, sym) 3483.20, 3328.91, U(C-F) 1225.55;[1] H NMR($CDCl_3$) δ: 4.23(S,4H –NH2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S, 2H –NH);6.722-6.700[d, J = 8.8Hz,4 H –

C(3,3',3",3'")] ;7.647-7.626 [d, J =8.4 Hz,4 H –C(4,4',4",4'")];7.332-7.300 [d, J =12.8 Hz,4 H –C(5,5',5",5")]; 7.261-7.230[d, J =12.4Hz,4 H –C(6,6',6",6")]; 3.752-3.717 [m,, J =14 Hz, 4H –C(7,7',8,8')];4.121-4.086 [m,2H –C(9,9')];[13] C- NMR ($CDCl_3$) δ : 181.03[C(1), C(1')] ; 105.39 [C(2), C(2')]; 152.31[C(3), C(3')]; 142.98[C(4), C(4')] ; 116.90-116.88 [C(5), C(5')]; 130.74-130.70[C(6), C(6')]; 129.60 [C(7), C(7')]; 152.71[C(8), C(8')]; 40.1 [C(9), C(9')]; 66.89 [C(10), C(10')]; 175.62 [C(11), C(11')]; 140.88 [C(12), C(12')]; 129.28-129.20 [C(13), C(13')]; 115.75-115.73 [C(14), C(14')]; 160.25 [C(15), C(15')];Anal.(%) for $C_{38}H_{30}F_2N_8O_2S_2$,Calcd.C, 62.28; H, 4.13; N,15.29;

Found: C,62.23; H, 4.11; N, 15.34.

3,3'-(4,4'-(3,6-dioxocyclohexa-1,4-diene-1,4-diyl)bis(azanediyl)bis(4,1-phenylene))bis(5-(4-chlorophenyl)-4,5-dihydro-1H-pyrazole-1-carbothioamide) (AQ8). Yield89%, mp.134-136 C° .IR (KBr pellet, cm $^{-1}$):U(C=Oquinone) 1635.43, U(S=O) 1289.11, U(C=N) 1589.06, U(C= C) 1602.32, U(C- Haromatic) 3058.87, U(C- Haliphatic) 2987.58-2891.12 U(NH2asym,sym) 3483.15, 3328.86, U(C-Cl) 1100.91;[1] H NMR($CDCl_3$) δ: 4.23(S,4H –NH2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S, 2H –NH);6.722-6.700[d, J =8.8 Hz,4 H –C(3,3',3",3'")] ;7.647-7.626 [d, J =8.44 Hz,4 H –C(4,4',4",4'")];7.311-7.299 [d, J =4.8 Hz,4 H –C(5,5',5",5")]; 7.201-7.191[d, J =4.00 Hz,4 H –C(6,6',6",6")]; 3.752-3.717 [m, J =14 Hz 4H –C(7,7',8,8')];4.121-4.086 [m, J =14 Hz,2H –C(9,9')];[13] C- NMR δ($CDCl_3$) : 181.03[C(1), C(1')] ; 105.39 [C(2), C(2')]; 152.31[C(3), C(3')]; 142.98[C(4), C(4')] ; 116.90-116.88 [C(5), C(5')]; 130.74-130.70[C(6), C(6')]; 129.60 [C(7), C(7')]; 152.71[C(8), C(8')]; 40.27 [C(9), C(9')]; 66.69 [C(10), C(10')]; 175.22 [C(11), C(11')]; 144.98 [C(12), C(12')]; 127.11-127.10 [C(13), C(13')]; 128.02-128.00 [C(14), C(14')]; 132.26 [C(15), C(15')];Anal.(%) for $C_{38}H_{30}Cl_2N_8O_2S_2$, Calcd. C,59.60; H,3.95; N, 14.63;found: C, 59.66; H: 4.04; N: 14.61.

3,3'-(4,4'-(3,6-dioxocyclohexa-1,4-diene-1,4-diyl)bis(azanediyl)bis(4,1-phenylene))bis(5-(4-bromophenyl)-4,5-dihydro-1H-pyrazole-1-carbothioamide) (AQ9). Yeild94%,mp.186-188;IR (KBr pellet, cm $^{-1}$):U(C=Oquinone) 1631.67, U(S=O) 1286.43, U(C=N) 1589.23, U(C= C) 1603.67, U(C- Haromatic) 3058.89, U(C- Haliphatic) 2981.50-2891.11 U(NH2asym, sym) 3492.18, 3328.60, U(C-Br) 1029.92;[1] H NMR($CDCl_3$) δ: 4.23(S,4H –NH2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S, 2H –NH);6.722-6.700[d, J = 8.8 Hz,4 H –C(3,3',3",3'")] ;7.647-7.626 [d, J =8.4 Hz,4 H –C(4,4',4",4'")];7.282-7.243 [d, J =15.6 Hz,4 H –C(5,5',5",5")]; 7.999-7.961[d, J =15.2Hz,4 H –C(6,6',6",6")]; 3.752-3.717[m, J =14Hz, 4H –C(7,7',8,8')];4.121-4.086 [m, J =14Hz, 2H –C(9,9')];[13] C- NMR($CDCl_3$) δ : 181.03[C(1), C(1')] ; 105.39 [C(2), C(2')]; 152.31[C(3), C(3')]; 142.98[C(4), C(4')] ; 116.90-116.88 [C(5), C(5')]; 130.74-130.70[C(6), C(6')]; 129.60 [C(7), C(7')]; 152.71[C(8), C(8')]; 40.28 [C(9), C(9')]; 66.89 [C(10), C(10')]; 175.52 [C(11), C(11')]; 145.38 [C(12), C(12')]; 127.33--127.30 [C(13), C(13')]; 131.67--131.6 [C(14), C(14')]; 121.95 [C(15), C(15')];Anal.(%) for $C_{38}H_{30}Br_2N_8O_2S_2$, Calcd. C,53.40;H,3.54; N, 13.11;found: . C, 53.37; H, 3.59; N,12.99.

3,3'-(4,4'-(3,6-dioxocyclohexa-1,4-diene-1,4-diyl)bis(azanediyl)bis(4,1-phenylene))bis(5-(4-nitrophenyl)-4,5-dihydro-1H-pyrazole-1-carbothioamide)(AQ10).Yield94%,mp187-189 C°;IR (KBr pellet, cm $^{-1}$):U(C=Oquinone) 1641.60, U(S=O) 1290.22, U(C=N) 1591.19, U(C= C) 1604.12, U(C- Haromatic) 3070.46, U(C- Haliphatic) 2984.67-28890.46, U(NH2asym,sym) 3483.20, 3384.84.1 H NMR(CDCl3) δ: 4.23(S,4H –NH2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S, 2H –NH);6.695-6.674[d, J =8.4 Hz,4 H –C(3,3',3",3'")] ;7.647-7.626 [d, J =8.4Hz,4 H –C(4,4',4",4'")];7.454-7.415 [d, J =15.6 Hz,4 H –C(5,5',5",5")]; 8.339-8.300[d, J = 15.6 Hz,4 H –C(6,6',6",6")]; 3.752-3.717[m, J =14 Hz, 4H –C(7,7',8,8')];4.121-4.086 [m, J =14 Hz, 2H –C(9,9')].[13] C- NMR ($CDCl_3$) δ : 181.03[C(1), C(1')] ; 105.39 [C(2), C(2')]; 152.31[C(3), C(3')]; 142.98[C(4), C(4')] ; 116.90-116.88 [C(5), C(5')]; 130.74-130.70[C(6), C(6')]; 129.60 [C(7), C(7')]; 152.71[C(8), C(8')]; 40.29 [C(9), C(9')]; 66.59 [C(10), C(10')]; 175.24 [C(11), C(11')]; 147.32 [C(12), C(12')]; 123.63-123.60 [C(13), C(13')]; 123.76-123.77 [C(14), C(14')];145.9 [C(15), C(15')]; Anal. (%) for$C_{38}H_{30}N_{10}O_6S_2$, Calcd. C,58.01; H,3.84; N,17.80;found: C, 57.93; H, 3.87; N, 17.74.

3,3'-(4,4'-(3,6-dioxocyclohexa-1,4-diene-1,4-diyl)bis(azanediyl)bis(4,1-phenylene))bis(5-p-tolyl-4,5-dihydro-1H-pyrazole-1-carbothioamide)(AQ11). Yeild91%,mp144-146C°;IR (KBr pellet, cm -1):U(C=Oquinone) 1627.81, U(S=O) 1290.29, U(C=N) 1569.95, U(C=C) 1595.02, U(C- Haromatic) 3026.10, U(C- Haliphatic) 2910.38-2852.52, U(NH2asym, sym) 3490.92,3346.27;[1] H NMR(CDCl3) δ: 4.23(S,4H –NH2

); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S, 2H –NH);6.722-6.700[d, J =8.8 Hz,4 H –C(3,3',3",3"')] ;7.647-7.626 [d, J =8.4 Hz,4 H –C(4,4',4",4"')];7.261-7.241 [d, J = 8 Hz,4 H –C(5,5',5",5")];7.321-7.299[d, J = 8.8 Hz,4 H –C(6,6',6",6")]; 3.752-3.717 [m, J = 14 Hz, 4H –C(7,7',8,8')];4.121-4.086 [m,, J = 14 Hz, 2H –C(9,9')];2.501[S,3H-CH_3];[13] C- NMR($CDCl_3$) $^{\delta}$: 181.03[C(1), C(1')] ; 105.39 [C(2), C(2')]; 152.31[C(3), C(3')]; 142.98[C(4), C(4')] ; 116.90-116.88 [C(5), C(5')]; 130.74-130.70[C(6), C(6')]; 129.60 [C(7), C(7')]; 152.71[C(8), C(8')]; 40.20 [C(9), C(9')]; 66.85 [C(10), C(10')]; 175.12 [C(11), C(11')]; 137.42 [C(12), C(12')]; 125.03-125.00 [C(13), C(13')]; 128.77-128.73 [C(14), C(14')]; 138.21 [C(15), C(15')]; 21.65[C(16), C(16')] ;Anal. (%) for $C_{40}H_{36}N_8O_2S_2$Calcd.C,66.28; H,5.01; N, 15.46;found: C, 66.22; H, 4.86; N,15.43.

3,3'-(4,4'-(3,6-dioxocyclohexa-1,4-diene-1,4-diyl)bis(azanediyl)bis(4,1-phenylene))bis(5-(4-hydroxyphenyl)-4,5-dihydro-1H-pyrazole-1-carbothioamide) (AQ12). Yeild90%,mp.135-136C°;IR (KBr pellet, cm -1):U(C=Oquinone) 1631.11, U(S=O) 1288.36, U(C=N) 1571.88, U(C= C) 1592.07, U(C-Haromatic) 3019.82, U(C-Haliphatic) 2938.27-2881.03, U(NH2asym, sym) 3490.90, 3354.93U(O- H) 3222.83;[1] H NMR ($CDCl_3$)$^{\delta}$: 4.23(S,4H –NH2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S, 2H –NH);6.814-6.811[d, J =1.2 Hz,4 H –C(3,3',3",3"')] ;7.875-7.873 [d, J =0.8Hz,4 H –C(4,4',4",4"')];7.151-7.147 [d, J =1.6Hz,4 H –C(5,5',5",5")];6.933-6.929[d, J =1.6 Hz,4 H –C(6,6',6",6")]; 3.752-3.717[m, J =14 Hz, 4H –C(7,7',8,8')];4.121-4.086 [m, J =14 Hz, 2H –C(9,9')];9.472[S,2H-C(11,11')];[13] C- NMR($CDCl_3$) $^{\delta}$: 181.03[C(1), C(1')] ; 105.39 [C(2), C(2')]; 152.31[C(3), C(3')]; 142.98[C(4), C(4')] ; 116.90-116.88 [C(5), C(5')]; 130.74-130.70[C(6), C(6')]; 129.60 [C(7), C(7')]; 152.71[C(8), C(8')]; 40.21 [C(9), C(9')]; 66.19 [C(10), C(10')]; 175.23 [C(11), C(11')]; 135.24 [C(12), C(12')]; 127.44127.42 [C(13), C(13')]; 115.29-115.27 [C(14), C(14')]; 138.21 [C(15), C(15')]; 21.65[C(16), C(16')] ;Anal. (%)for $C_{38}H_{32}N_8O_4S_2$, Calcd. C,65.51; H,4.63; N,16.08;found : C, 65.41; H, 4.69; N, 16.11.

2,5-bis(4-(6-(4-fluorophenyl)-2-hydroxypyrimidin-4-yl)phenylamino)cyclohexa-2,5-diene-1,4-dione(AQ13) . Yeild 88% ,mp.176-177C°.IR (KBr pellet, cm $^{-1}$):U(C=O $_{quinone}$) 1636.59, U(C-O) 1284.50, U(C=N) 1598.74, U(C= C) 1600.39, U(C- $H_{aromatic}$) 3058.89, , U($NH_{2asym,sym}$) 3446.77, 3446.27, U(C-F) 1220.86. [1] H NMR($CDCl_3$) $^{\delta}$: 4.23(S,4H –NH_2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S , 2H –NH);6.892-6.872[d, *J* = 8 Hz,4 H –C(3,3',3",3"')] ;7.512-7.492 [d, *J* = 8 Hz,4 H –C(4,4',4",4"')];8.147-8.116 [d, *J* = 12.4 Hz,4 H –C(5,5',5",5")];7.373-7.342[d, *J* =12.4 Hz,4 H –C(6,6',6",6")]; 7.632 [S, 2H –C(7,7')];11.626 [S,2H –O(8,8')]; [13] C- NMR($CDCl_3$) $^{\delta}$: 181.6 [C(1) , C(1')] ; 109.37 [C(2) , C(2')]; 152.31 [C(3) , C(3')]; 142.20 [C(4) , C(4')] ; 114.48-114.50 [C(5) , C(5')]; 129.32-129.31 [C(6) , C(6')]; 125.98 [C(7) , C(7')]; 162.35 [C(8) , C(8')]; 93.27 [C(9) , C(9')]; 155.32 [C(10) , C(10')]; 160.39 [C(11) , C(11')]; 131.14 [C(12) , C(12')]; 130.31-130.30 [C(13) , C(13')]; 115.95-115.94 [C(14) , C(14')]; 162.14 [C(15) , C(15')];Anal.(%) for $C_{38}H_{24}F_2N_6O_4$,Calcd. C, 68.46; H, 3.63; N, 12.61;found: C, 68.35; H, 3.57; N, 12.63.

2,5-bis(4-(6-(4-fluorophenyl)-2-mercaptopyrimidin-4-yl)phenylamino)cyclohexa-2,5-diene-1,4-dione(AQ14). Yeild 91% ,mp.128-130;IR (KBr pellet, cm $^{-1}$):U(C=$O_{quinone}$) 1636.88, U(C-N) 1292.22, U(C= N) 1544.88, U(C= C) 1600.81, U(C- $H_{aromatic}$) 3234.40, , U(NH) 3357.84, U(C-F) 1220.86, U(S-H) 2360.71,U(C-F) 1220.86. [1] H NMR($CDCl_3$) $^{\delta}$: 4.23(S,4H –NH_2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S , 2H –NH);6.892-6.872[d, *J* =8 Hz,4 H –C(3,3',3",3"')] ;7.512-7.492 [d, *J* =8 Hz,4 H –C(4,4',4",4"')];8.147-8.116 [d, *J* =12.4 Hz,4 H –C(5,5',5",5")];7.342-7.334[d, *J* =12.4 Hz,4 H –C(6,6',6",6")]; 8.479 [S, 2H –C(7,7')];12.126 [S,2H –S(8,8'; [13] C- NMR($CDCl_3$) $^{\delta}$:181.39 [C(1) , C(1')] ; 109.37 [C(2) , C(2')]; 152.31 [C(3) , C(3')]; 142.20 [C(4) , C(4')] ; 114.48-114.50 [C(5) , C(5')]; 129.32-129.31 [C(6) , C(6')]; 125.98 [C(7) , C(7')]; 169.63 [C(8) , C(8')]; 109.32 [C(9) , C(9')]; 166.32 [C(10) , C(10')]; 181.06 [C(11) , C(11')]; 131.14 [C(12) , C(12')]; 130.31-130.30 [C(13) , C(13')]; 115.95-115.94 [C(14) , C(14')]; 162.14 [C(15) , C(15')] ;Anal (%) for C38H24F2N6O4 ,Calcd. C, 65.32; H, 3.46; N, 12.03;found: C, 65.41; H,3.41; N, 12.00.

2,5-bis(4-(6-(4-chlorophenyl)-2-hydroxypyrimidin-4-yl)phenylamino)cyclohexa-2,5-diene-1,4-dione(AQ15). Yeild86%,mp97-99C°,IR (KBr pellet, cm $^{-1}$):U(C=$O_{quinone}$) 1635.59, U(C-N) 1273.51, U(C= N) 1573.34, U(C= C) 1600.71, U(C- $H_{aromatic}$) 3054.83, , U(NH) 3443.76, U(C-Cl) 1101.98, U(O-H) 3342.24;[1] H NMR($CDCl_3$) $^{\delta}$: 4.23(S,4H –NH_2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S , 2H –NH);6.692-6.672 d, *J* =8 Hz,4 H –C(3,3',3",3"')] ;7.526-7.506 [d, *J* =8 Hz,4 H –C(4,4',4",4"')];7.987-7.956 [d, *J* =12.4 Hz,4 H –C(5,5',5",5")];7.558-7.527[d, *J* =12.4 Hz,4 H –C(6,6',6",6")]; 7.632 [S, 2H –C(7,7')];11.337 [S,2H –O(8,8')]; [13]

C- NMR($CDCl_3$) δ :181.06 [C(1) , C(1')] ; 101.30 [C(2) , C(2')]; 152.31 [C(3) , C(3')]; 142.20 [C(4) , C(4')] ; 114.48-114.50 [C(5) , C(5')]; 128.41128.39 [C(6) , C(6')]; 125.69 [C(7) , C(7')]; 162.35 [C(8) , C(8')]; 93.27 [C(9) , C(9')]; 155.32 [C(10) , C(10')]; 160.39 [C(11) , C(11')]; 133.19 [C(12) , C(12')]; 129.31-129.30 [C(13) , C(13')]; 129.33-129.32 [C(14) , C(14')]; 135.14 [C(15) , C(15')];Anal. (%) forC38H24Cl2N6O4 Calcd . C, 65.24; H, 3.46; N, 12.01,found: C, 65.23; H, 3.51; N, 11.98.

2,5-bis(4-(6-(4-chlorophenyl)-2-mercaptopyrimidin-4-yl)phenylamino)cyclohexa-2,5-diene-1,4-dione(AQ16). Yeild 81% ,mp.176-177C° ,IR (KBr pellet, cm $^{-1}$):U(C=$O_{quinone}$) 1635.74, U(C-N) 1285.62, U(C= N) 1596.95, U(C= C) 1606.59, U(C- $H_{aromatic}$) 3066.43, , U(NH) 3357.84, U(S-H) 2553.58,U(C-Cl) 1108.34.. ^{1} H NMR($CDCl_3$) δ: 4.23(S,4H –NH_2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S , 2H –NH);6.692-6.672 d, *J* =8 Hz,4 H –C(3,3',3",3"')] ;7.526-7.506 [d, *J* =8 Hz,4 H –C(4,4',4",4"')];7.987-7.956 [d, *J* =12.4Hz,4 H –C(5,5',5",5")];7.558-7.527[d, *J* =12.4Hz,4 H –C(6,6',6",6")]; 8.158 [S, 2H –C(7,7')];12.187 [S,2H –S(8,8')]; ^{13} C- NMR($CDCl_3$) δ :181.09 [C(1) , C(1')] ; 101.33 [C(2) , C(2')]; 152.31 [C(3) , C(3')]; 142.20 [C(4) , C(4')] ; 114.48-114.50 [C(5) , C(5')]; 128.41-128.39 [C(6) , C(6')]; 125.63 [C(7) , C(7')]; 167.98 [C(8) , C(8')]; 109.37 [C(9) , C(9')]; 164.94 [C(10) , C(10')]; 181.06 [C(11) , C(11')]; 133.19 [C(12) , C(12')]; 129.31-129.30 [C(13) , C(13')]; 129.33-129.32 [C(14) , C(14')]; 135.14 [C(15) , C(15')];Anal.(%) for $C_{38}H_{24}Cl_2N_6O_2S_2$,Calcd. C, 62.38; H, 3.31; N, 11.49;found : . C, 65.23; H, 3.28; N, 11.52.

2,5-bis(4-(6-(4-bromophenyl)-2-hydroxypyrimidin-4-yl)phenylamino)cyclohexa-2,5-diene-1,4-dione(AQ17). Yeild 91% ,mp.98-100C°,IR (KBr pellet, cm $^{-1}$):U(C=$O_{quinone}$) 1631.55, U(C-N) 1284.50, U(C= N) 1591.54, U(C= C) 1600.13, U(C- $H_{aromatic}$) 3054.87, , U(NH) 3447.73, U(CBr) 1025.91, U(O-H) 3464.27; ^{1} H NMR($CDCl_3$) δ: 4.23(S,4H –NH_2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S , 2H –NH);7.294-7.268 d, *J* =10.4Hz,4 H –C(3,3',3",3"')] ;7.598-7.572[d, *J* =10.4 Hz,4 H –C(4,4',4",4"')];7.692-7.656 [d,*J*=14.4Hz,4 H –C(5,5',5",5")];7.647-7.6612[d,*J*=14.0Hz,4H–C(6,6',6",6")];7.598 [S, 2H –C(7,7')];11.626 [S,2H –O(8,8')]; ^{13} C-NMR($CDCl_3$) δ : 181.06 [C(1) , C(1')] ; 101.34 [C(2) , C(2')]; 152.31 [C(3) , C(3')]; 142.20 [C(4) , C(4')] ; 114.48-114.50 [C(5) , C(5')]; 93.27 [C(9) , C(9')]; 155.32 [C(10) , C(10')]; 160.39 [C(11) , C(11')]; 134.11 [C(12) , C(12')]; 129.32-129.31 [C(13) , C(13')]; 133.54-133.52 [C(14) , C(14')]; 123.34 [C(15) , C(15')] ;Anal.(%) for $C_{38}H_{24}Br_2N_6O_4$,Calcd.C, 57.89; H, 3.07; N, 10.66;found: C,57.92; H, 3.13; N,10.60.

2,5-bis(4-(6-(4-bromophenyl)-2-mercaptopyrimidin-4-yl)phenylamino)cyclohexa-2,5-diene-1,4-dione(AQ18). Yeild 85% ,mp.142-144C° .IR (KBr pellet, cm $^{-1}$):U(C=$O_{quinone}$) 1631.61, U(C-N) 1280.65, U(C= N) 1591.72, U(C= C) 1606.59, U(C- $H_{aromatic}$) 3069.01, , U(NH) 3352.88, U(S-H) 2553.55,U(C-Br) 1091.12. . ^{1} H NMR($CDCl_3$) δ: 4.23(S,4H –NH_2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S , 2H –NH);7.294-7.268 d, *J* = 10.4 Hz,4 H –C(3,3',3",3"')] ;7.598-7.572 [d, *J* =10.4 Hz,4 H –C(4,4',4",4"')];7.692-7.656 [d, *J* =14.4 Hz,4 H –C(5,5',5",5")];7.647-7.6612[d, *J* =14.0 Hz,4 H –C(6,6',6",6")]; 8.402 [S, 2H –C(7,7')];12.124 [S,2H –S(8,8')];^{13} C- NMR δ : 181.09 [C(1) , C(1')] ; 101.18 [C(2) , C(2')]; 152.31 [C(3) , C(3')]; 142.20 [C(4) , C(4')] ; 114.48-114.50 [C(5) , C(5')]; 128.91-128.90 [C(6) , C(6')]; 125.54 [C(7) , C(7')]; 169.50 [C(8) , C(8')]; 109.37 [C(9) , C(9')]; 165.38 [C(10) , C(10')]; 181.06C(11) , C(11')]; 134.11 [C(12) , C(12')]; 129.32-129.31 [C(13) , C(13')]; 133.54-133.52 [C(14) , C(14')]; 123.34 [C(15) , C(15')];Anal. (%) for $C_{38}H_{24}Br_2N_6O_2S_2$,Calcd. C, 55.62; H, 2.95; N, 10.24;found: C, 55.65; H, 3.01; N, 10.22.

2,5-bis(4-(2-mercapto-6-(4-nitrophenyl)pyrimidin-4-yl)phenylamino)cyclohexa-2,5-diene-1,4-dione(AQ19). Yeild 93% ,mp.111-113C° ,IR (KBr pellet, cm $^{-1}$):U(C=$O_{quinone}$) 1639.52, U(C-N) 1290.29, U(C= N) 1591.16, U(C= C) 1600.38, U(C- $H_{aromatic}$) 3070.46, , U(NH) 3483.20, 3384.84 U(S-H) 2228.62, 1091.12; ^{1} H NMR($CDCl_3$) δ: 4.23(S,4H –NH_2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S , 2H –NH);6.722-6.700 d, *J* =8.8 Hz,4 H –C(3,3',3",3"')] ;7.519-7.497 [d, *J* =8.8 Hz,4 H –C(4,4',4",4"')];8.043-8.004 [d, *J* =15.6Hz,4 H –C(5,5',5",5")];8.237-8.198[d, *J* =15.6Hz,4 H –C(6,6',6",6")];8.543 [S, 2H –C(7,7')];12.340[S,2H –S(8,8')] ; ^{13} C- NMR δ($CDCl_3$) : 181.06 [C(1) , C(1')] ; 101.37 [C(2) , C(2')]; 152.31 [C(3) , C(3')]; 142.20 [C(4) , C(4')] ; 114.48-114.50 [C(5) , C(5')]; 129.35-129.33 [C(6) , C(6')]; 125.34 [C(7) , C(7')]; 165.65 [C(8) , C(8')]; 109.87 [C(9) , C(9')]; 164.62 [C(10) , C(10')]; 181.03C(11) , C(11')]; 141.12 [C(12) , C(12')]; 126.51-126.50 [C(13) , C(13')]; 124.34--124.30 [C(14) , C(14')]; 149.64 [C(15) , C(15')];Anal.(%) for $C_{38}H_{24}N_8O_6S_2$,Calcd. C, 63.33; H, 3.36; N: 15.55;found : C, 63.35; H, 3.26; N: 15.51.

2,5-bis(4-(2-hydroxy-6-(4-nitrophenyl)pyrimidin-4-yl)phenylamino)cyclohexa-2,5-diene-1,4-dione(AQ20) . Yeild 88% ,mp.148-151C°,IR (KBr pellet, cm $^{-1}$):U(C=$O_{quinone}$) 1638.74, U(C-N) 1292.22, U(C= N) 1544.88, U(C= C) 1600.81, U(C- $H_{aromatic}$) 3056.40, , U(NH) 3378.05, 3234.40, U(O-H) 2360.71; ^{1} H NMR($CDCl_3$) $^{\delta}$: 4.23(S,4H –NH_2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S , 2H –NH);6.722-6.700 d, *J* =8.8 Hz,4 H –C(3,3',3",3'")] ;7.519-7.497 [d, *J* =8.8 Hz,4 H –C(4,4',4",4'")];8.043-8.004 [d, *J* = 15.6 Hz,4 H –C(5,5',5",5")];8.237-8.198[d, *J* =15.6Hz,4 H –C(6,6',6",6")];8.543 [S, 2H –C(7,7')];11.340[S,2H –O(8,8')];^{13} C-NMR($CDCl_3$) $^{\delta}$: 181.8 [C(1) , C(1')] ; 101.37 [C(2) , C(2')]; 152.31 [C(3) , C(3')]; 142.20 [C(4) , C(4')] ; 114.48-114.50 [C(5) , C(5')]; 129.35-129.33 [C(6) , C(6')]; 125.34 [C(7) , C(7')]; 162.35 [C(8) , C(8')]; 93.27 [C(9) , C(9')]; 155.32 [C(10) , C(10')]; 160.39C(11) , C(11')]; 141.12 [C(12) , C(12')]; 126.51-126.50 [C(13) , C(13')]; 124.34--124.30 [C(14) , C(14')]; 149.64 [C(15) , C(15')] ;Anal. (%) for $C_{38}H_{24}N_8O_8$,Calcd.C, 60.63; H, 3.21; N, 14.89;found: C,60.53; H, 3.22; N, 14.88.

2,5-bis(4-(2-hydroxy-6-p-tolylpyrimidin-4-yl)phenylamino)cyclohexa-2,5-diene-1,4-dione: (AQ21). Yeild 79% ,mp187-189C°,IR (KBr pellet, cm $^{-1}$):U(C=$O_{quinone}$) 1627.81, U(C-N) 1290.22, U(C= N) 1569.92, U(C= C) 1595.02, U(C- $H_{aromatic}$) 3026.11, U(C- $H_{aliphatic}$) 2910.30, 2852.57, U(NH) 3440.77, 3234.40, U(O-H) 3461.27;^{1} H NMR($CDCl_3$) $^{\delta}$: 4.23(S,4H –NH_2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S , 2H –NH);6.722-6.700 d, *J* =8.8 Hz,4 H –C(3,3',3",3'")] ;7.512-7.7.492 [d, *J* =8 Hz,4 H –C(4,4',4",4'")];7.671-7.632 [d, *J* =15.6 Hz,4 H –C(5,5',5",5")];7.294-7.255[d, *J* =15.6 Hz,4 H –C(6,6',6",6")];7.671 [S, 2H –C(7,7')];11.342[S,2H –O(8,8')]; 2.337[S,3H-C(10,10')]; ^{13} C- NMR($CDCl_3$) $^{\delta}$: 181.06 [C(1) , C(1')] ; 101.37 [C(2) , C(2')]; 152.31 [C(3) , C(3')]; 142.20 [C(4) , C(4')] ; 114.48-114.50 [C(5) , C(5')]; 129.35-129.33 [C(6) , C(6')]; 125.51 [C(7) , C(7')]; 162.35 [C(8) , C(8')]; 93.27 [C(9) , C(9')]; 155.32 [C(10) , C(10')]; 160.39C(11) , C(11')]; 131.11 [C(12) , C(12')]; 123.01-123.00 [C(13) , C(13')]; 129.32-129.31 [C(14) , C(14')]; 132.54 [C(15) , C(15')] ; 23.87[C(16) , C(16')];Anal. (%) for $C_{40}H_{30}N_6O_4$,Calcd. C, 72.94; H, 4.59; N, 12.76;found: C,72.96; H, 4.52; N, 12.79.

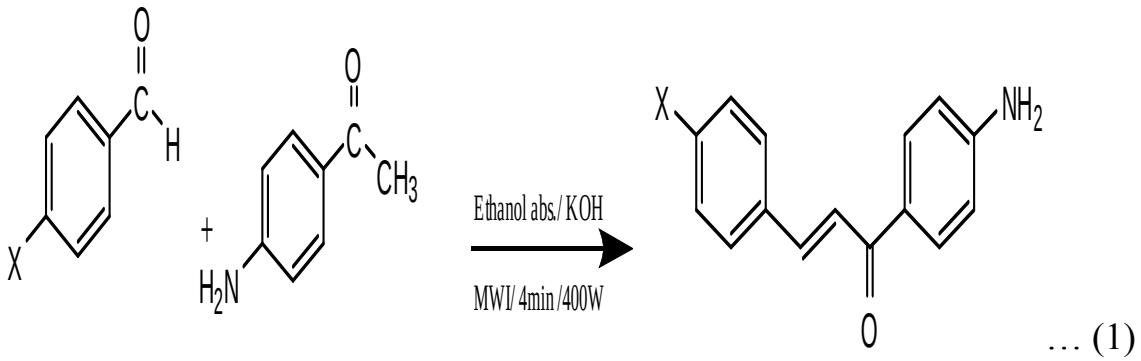
2,5-bis(4-(2-mercapto-6-p-tolylpyrimidin-4-yl)phenylamino)cyclohexa-2,5-diene-1,4-dione(AQ22) Yeild 92% ,mp.(173-175), IR (KBr pellet, cm $^{-1}$):U(C=$O_{quinone}$) 1625.88, U(C-N) 1292.22, U(C= N) 1544.88, U(C= C) 1600.81, U(C- $H_{aromatic}$) 3019.05, U(C- $H_{aliphatic}$) 2911.42, 2867.09, U(NH) 3461.99, 3344.34 U(S-H) 2360.71; ^{1} H NMR($CDCl_3$) $^{\delta}$: 4.23(S,4H –NH_2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S , 2H –NH);6.722-6.700 d, *J* =8.8Hz,4 H –C(3,3',3",3'")] ;7.512-7.7.492 [d, *J* =8 Hz,4 H –C(4,4',4",4'")];7.671-7.632 [d, *J* = 15.6 Hz,4 H –C(5,5',5",5")];7.294-7.255[d, *J* =15.6 Hz,4 H –C(6,6',6",6")];8.181 [S, 2H –C(7,7')];12.184[S,2H –S(8,8')]; 2.337[S,3H-C(10,10')]; ^{13} C- NMR($CDCl_3$) $^{\delta}$: 181.60 [C(1) , C(1')] ; 101.01 [C(2) , C(2')]; 152.31 [C(3) , C(3')]; 142.20 [C(4) , C(4')] ; 114.48-114.50 [C(5) , C(5')]; 129.35-129.33 [C(6) , C(6')]; 125.51 [C(7) , C(7')]; 165.36 [C(8) , C(8')]; 109.37 [C(9) , C(9')]; 165.32 [C(10) , C(10')]; 181.5C(11) , C(11')]; 132.54 [C(12) , C(12')]; 123.01-123.00 [C(13) , C(13')]; 129.32-129.31 [C(14) , C(14')]; 131.11 [C(15) , C(15')] ; 23.87[C(16) , C(16')];Anal.(%) for$C_{40}H_{30}N_6O_2S_2$,Calcd. C, 69.54; H, 4.38; N, 12.17;found: C,69.56; H, 4.30; N, 12.19.

2,5-bis(4-(2-hydroxy-6-(4-hydroxyphenyl)pyrimidin-4-yl)phenylamino)cyclohexa-2,5-diene-1,4-dione(AQ23) . Yeild 95% ,mp.129-131C°,IR (KBr pellet, cm $^{-1}$):U(C=$O_{quinone}$) 1630.67, U(C-N) 1290.35, U(C= N) 1511.06, U(C= C) 16006.81, U(C- $H_{aromatic}$) 3013.72, , U(NH) 3340.88, 3217.93, U(O-H) Me.Wi NH_2. . ^{1} H NMR $^{\delta}$: 4.23(S,4H –NH_2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S , 2H –NH);6.722-6.700 d, *J* = Hz,4 H –C(3,3',3",3'")] ;7.503-7.481 [d, *J* = Hz,4 H –C(4,4',4",4'")];7.416-7.378 [d, *J* = Hz,4 H –C(5,5',5",5")];6.843-6.804[d, *J* = Hz,4 H –C(6,6',6",6")];7.615 [S, 2H –C(7,7')];11.339[S,2H –O(8,8')]; 9.386[S,2H-O(10,10')];^{13} C- NMR ($CDCl_3$) $^{\delta}$: 181.60 [C(1) , C(1')] ; 109.37 [C(2) , C(2')]; 152.31 [C(3) , C(3')]; 142.20 [C(4) , C(4')] ; 114.48-114.50 [C(5) , C(5')]; 129.35-129.33 [C(6) , C(6')]; 125.51 [C(7) , C(7')]; 162.35 [C(8) , C(8')]; 93.27 [C(9) , C(9')]; 155.32 [C(10) , C(10')]; 160.39C(11) , C(11')]; 128.74 [C(12) , C(12')]; 128.42-128.40 [C(13) , C(13')]; 116.39-116.38 [C(14) , C(14')]; 158.36 [C(15) , C(15')];Anal.(%) for $C_{38}H_{26}N_6O_6$Calcd.C: 68.88; H, 3.95; N, 12.68;found: 68.78; H, 3.95; N, 12.77.

2,5-bis(4-(6-(4-hydroxyphenyl)-2-mercaptopyrimidin-4-yl)phenylamino)cyclohexa-2,5-diene-1,4-dione(AQ24). Yeild92% ,mp.164-166C° .IR (KBr pellet, cm $^{-1}$):U(C=$O_{quinone}$) 1630.19, U(C-N) 1290.29, U(C= N) 1571.88, U(C= C) 1595.02, U(C- $H_{aromatic}$) 3004.95, U(C- $H_{aliphatic}$) 2911.42, 2867.09, U(NH)

3340.48, 3213.19,U(S-H) 2601.79 U(O-H) Me.Wi NH_2; ^{1}H NMR ($CDCl_3$)$^{\delta}$: 4.23(S,4H –NH_2); 6.306-6.6332(S,2H –C(1,1') ; 2.301(br. S , 2H –NH);6.722-6.700 d, *J* =8.8 Hz,4 H –C(3,3',3",3'")] ;7.503-7.481 [d, *J* =8.8 Hz,4 H –C(4,4',4",4'")];7.416-7.378 [d, *J* =15.2 Hz,4 H –C(5,5',5",5")];6.843-6.804[d, *J* = 15.6 Hz,4 H –C(6,6',6",6")];8.435 [S, 2H –C(7,7')];12.339[S,2H –O(8,8')]; 9.386[S,2H-O(10,10')]; ^{13} C- NMR($CDCl_3$) $^{\delta}$: 181.62 [C(1) , C(1')] ; 101.08 [C(2) , C(2')]; 152.31 [C(3) , C(3')]; 142.20 [C(4) , C(4')] ; 114.48-114.50 [C(5) , C(5')]; 129.35-129.33 [C(6) , C(6')]; 125.51 [C(7) , C(7')]; 165.25 [C(8) , C(8')]; 109.37 [C(9) , C(9')]; 165.12 [C(10) , C(10')]; 181.5C(11) , C(11')]; 128.74 [C(12) , C(12')]; 128.42-128.40 [C(13) , C(13')]; 116.39-116.38 [C(14) , C(14')]; 158.36 [C(15) , C(15')];Anal.(%) for $C_{38}H_{26}N_6O_4S_2$Calcd. C, 65.69; H, 3.77; N, 12.10;found: C, 65.66; H, 3.70; N, 12.00.

A series ofchalcones were prepared according to Clasien Schmidt condensation, of variously substituted aromatic aldehydes with Para amino acetophenone by using base catalyzed, under microwave irradiation to give corresponding chalcones. The reactions were monitored for its completion by TLC at 30 seconds intervals for (1-6) minutes depending on substituent's of benzyldehyde.The reaction is presented inequation 1.[17]



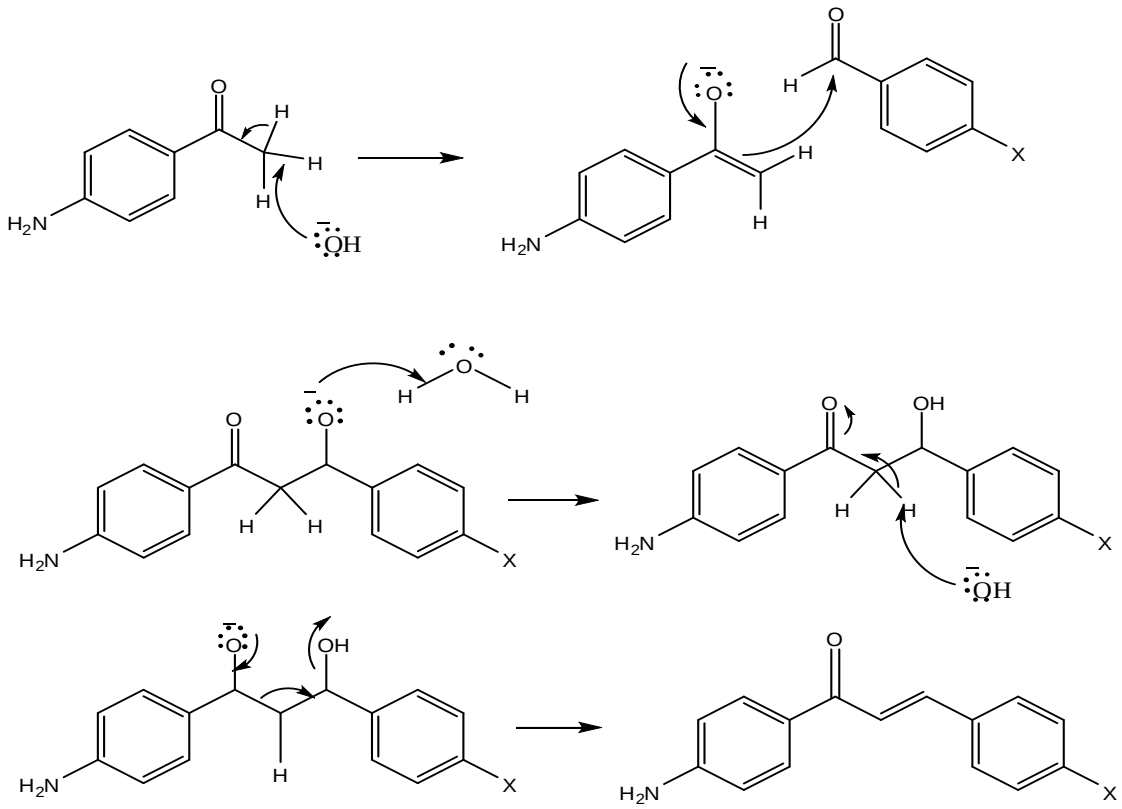
… (1)

X: F,Cl,Br,NO_2,OH,CH_3

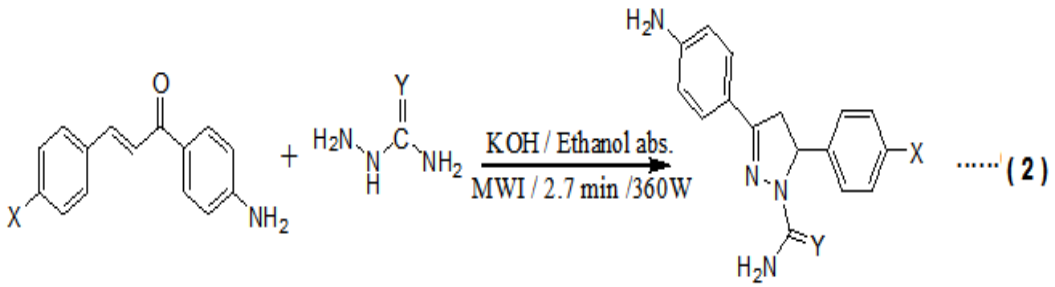
The synthesized chalcones showed different yields depending on the substituent groups as they were electron withdrawing group or electron donating group. Aldehydes with electron donating groups led to increasing the electron density on the carbon atom of carbonyl group, resulting in enhancing their electronic properties which subsequently decreasing the yield of the products. In contrast those with electron withdrawing groups resulted in increasing the yield as the electron densities on carbon atom of carbonyl were decreased.

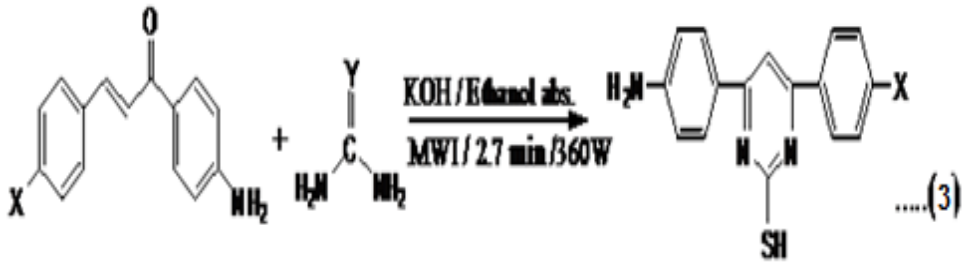
The mechanism of reaction can be explained in scheme (1) which showed the nucleophlic attack of emulateanion at carbon atom of carbonyl related to benzyldehyde with elimination of water at end of reaction.

Then these chalcones were mixed with (semicarbazide, thiosemicarbized,urea,thiourea) respectively with equal molar in presence of KOH, the mixture dissolved in absolute ethanol and irradiated in microwave as shown in equation (2) .it was found that the yield of products related to the ability of substituent group on drawing electrons.

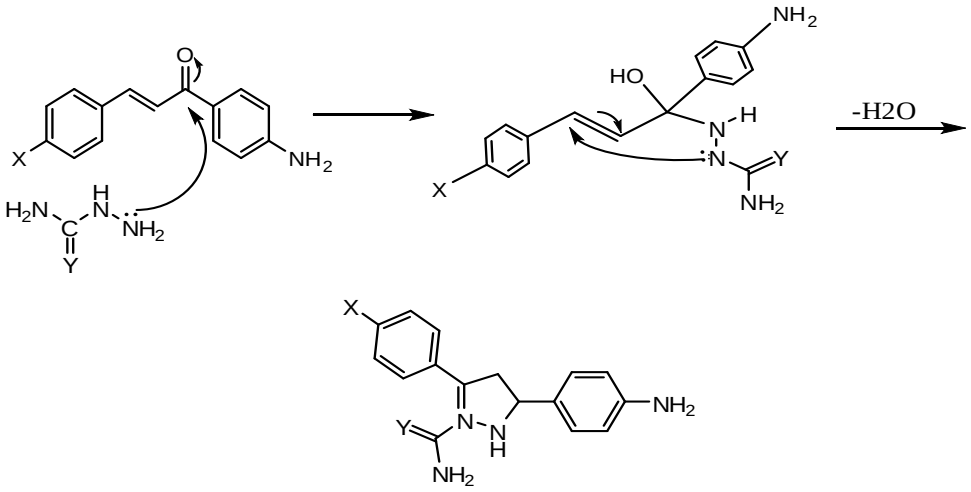


Scheme (1): mechanism of chalcone synthesis

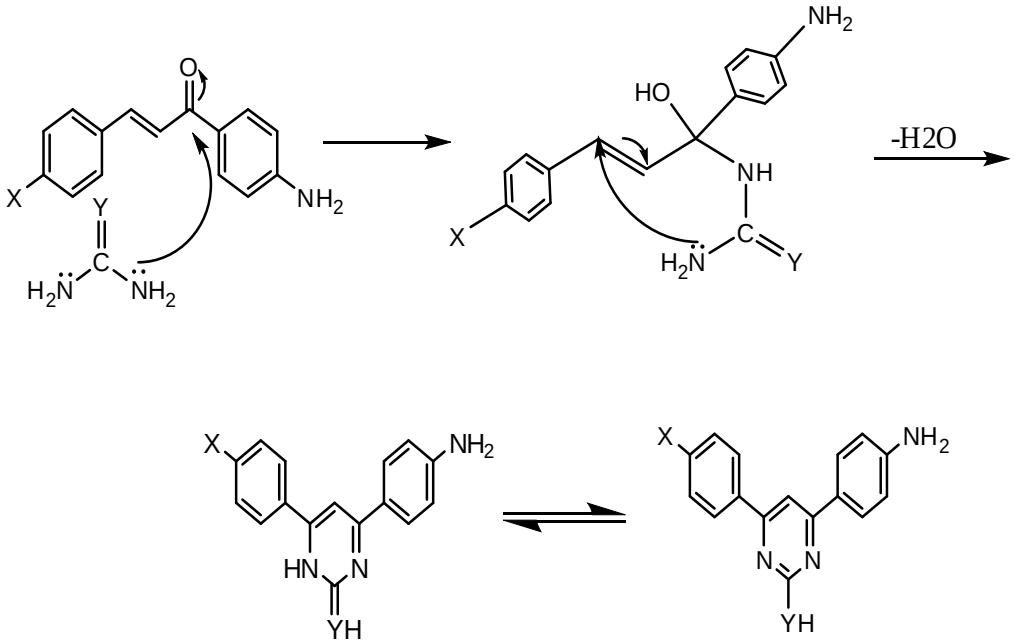




The mechanism of reactions[18], [19] can be explained by means of nucleophlic attack of amino group related to (semicrbizide, thiosemicarbizide) followed by cyclization.as described in scheme 2,3 respectively :

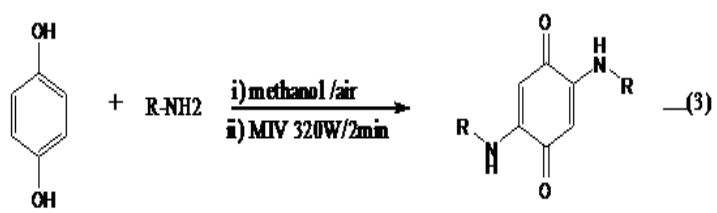


Scheme (2): mechanism of pyrazole synthesis

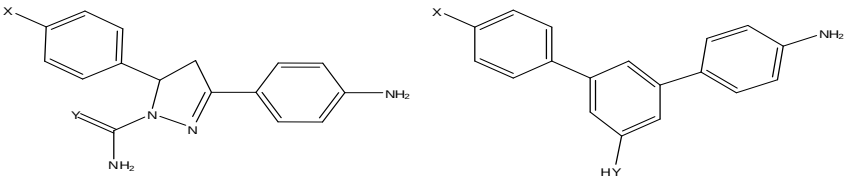


Scheme (3)

To synthesis 2,5-bis aminoquinonederivetives, (10ml) Methanol solution of amine (hetrocyclic series)(2.3 mmole) was added to methanol solution (10 ml) of hydroquinones (0.05 mmole).This mixture was irradiated at (320w) for two minutes .the solution was evaporated in room temperature then product was purified by recrystalization from methanol to obtain long crystals with different colors (red, orange, yellow) .As displayed in equation(3)

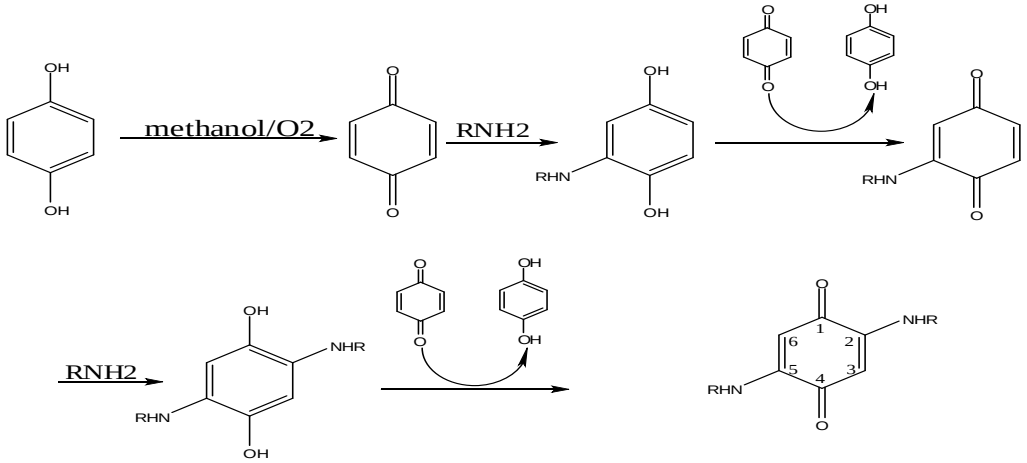


R:



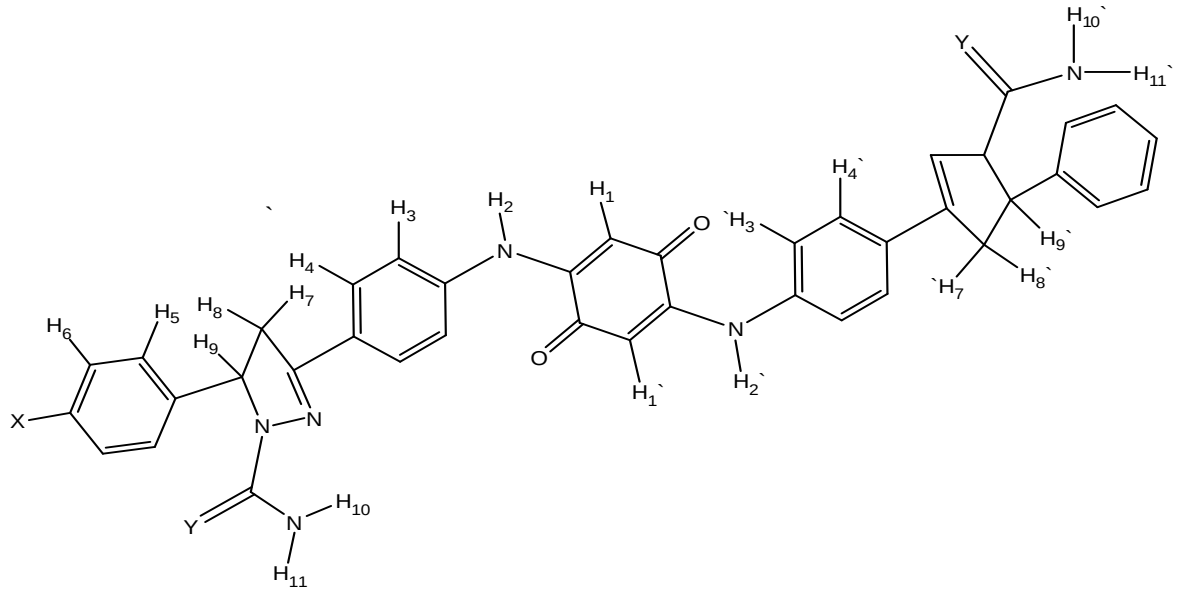
X: F,Cl,Br,NO2,OH,CH3
Y: S,O

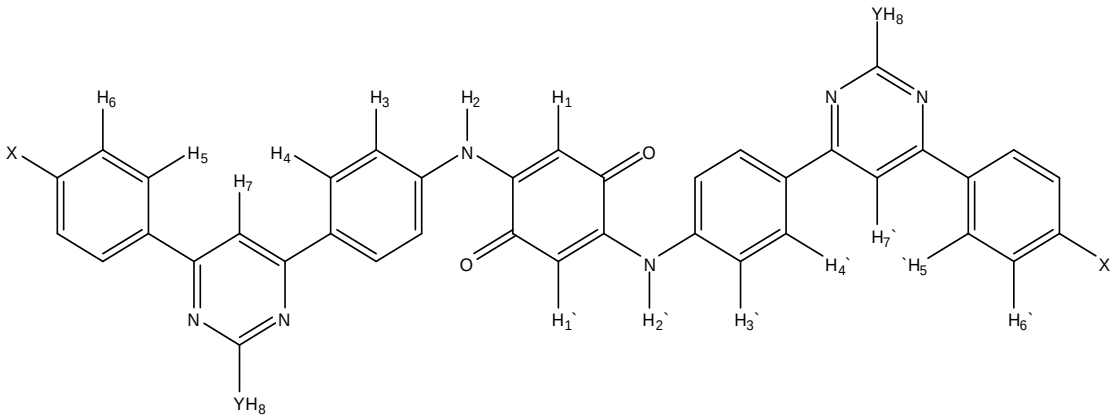
Mechanism of reaction [19] can be explained by nucleophlic attack of amino group according to 1, 4- addition (Michael addition) or conjugate addition.as illustrated in scheme (3).



Scheme (3): mechanism of 2,5Diaaminoquinone derivative synthesis

The structure of final products can be illustrated in the following structures:





X: -F, -Cl, -Br, -NO2, -OH, -CH3 . Y: OH, SH

The purity of compounds was determined by TLC and elemental analysis. Spectral data(IR,[1] H NMR,[13] C NMR) of all the newly synthesized compounds were in full agreement with proposed structures.

The infrared of all aminoquinone derivatives have common strong to medium absorption in the region (1637-1641cm^{-1}) due to the (C=O) stretching which they were characteristic to quinone moiety .peaks with variable intensity appeared in the range (3100-3050 cm^{-1}) were accounted to aromatic stretching vibration while weak bands were observed in the range (2856-2928cm^{-1}) related to (C-H) , (CH_2) , (CH_3) groups .stretching of primary and secondary amine as well as alcoholic groups bands were appeared in the range (3310-3485 cm^{-1}).tow strong peaks indicate aromatic double bond (C=C) stretching (1600- 1590 cm^{-1}) and (1471- 1479 cm^{-1}) .

It was found from spectra that carbonyl group absorption correlated with the electronic properties of substituent whether it was electron with drawing groups or electron donating groups, as described in the following arrangement :

-F > -Cl> -Br >

Their absorption were :(163.59 cm-1) ,(1635.74 cm-1) ,(1631.67 cm-1) respectively.

-OH > -CH_3

their absorption were (1630.67 cm-1), (1627.81 cm-1) respectively.

In the region (1220 cm-1) ,(1109cm-1) ,(1092cm-1) strong peaks appeared related to stretching of (C-F) ,(C-Cl) ,(C- Br) respectively.

^{1}H NMR of all spectra displayed strong single peak at (6.332- 6.300) ppm and medium single peak at (4.203-4.187) ppm related quinoniod protons (H1,H1`) and secondary amine protons (H2,H2`)respectively .

Multiple peaks appeared at the reagion (3.709-4.121) ppm related to heterocyclic protons(H7,H8,H9) in the compounds (AQ1- AQ12).Singlet broud peak appeared at (2.301)ppm due to primary amine protons (H10,H11) in the compounds (AQ1- AQ6) but the same peak shifted to lower magnetic field (4.752)ppm.The aromatic system classified into two groups A and B .protons of B ring(H5,H6) showed AX system in all compounds while protons of A ring showed both AX and AB system depending on the substituent's on para position .

Ratio of chemical shift differences to coupling constant was calculated ,and found to be smaller than 10 for the compounds (AQ1,AQ2,AQ5,AQ7,AQ8,AQ11,AQ17,AQ18) which indictes AB system for these compounds while others displayed AX system scince value of ( ) more than 10.^{13}C NMR related to All spectra showed peaks at (181.8 -181.06),(109.37 – 101.18), (152.31),(142.20), (114.48-114.50),(129.32-129.31) - (128.41-128.39) ,(125.69) related to carbon (C_1 ,C_2 , C_3 , C_4 , C_5 ,C_6 ,C_7) respectively.

It was observed that chemical shift for C_{12} decreased by the presence of electron with drawing groups in para position.[20]-[24].

Conclusion

The 2,5-Bis(heteroamino)-1,4-benzoquinone derivatives , were synthesized successfully throughout aerobic oxidation of hydroquinone followed by conjugate addition of primary amine according to Michael addition protocol under microwave irradiation . All spectral data improved the full agreement of the chemical structure for synthesized compounds.

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