Chapter two

2. Material and Methods

2.1 Materials

2.1.1. Chemicals:

- Acetone, 99.5%, Lab Tech, United States.
- Acetophenone, 99%, Anisaldehyde, 98%, Benzaldehyde, 98.5%, Noirtoacetophenone, 99%, Cinnamaldehyde, 99%, *p*-bromoacetophenone, 98%, *p*-dimethyl amino benzaldehyde, 98%, synthesis grade, Loba Chemie, india.
- Duterated Chloroform, 98%, Sigma Aldrich, United States.
- Ethanol, 99.1%, absolute, Bios Europe, United Kingdom.
- Ethanol, 97%, commercial local product of Al-watania Company.
- Furfural, 98%, for synthesis, Loba Chemie, india.
- Guanidine hydrochloride, 99.5%, extra pure, Fisher Scientific, product of China.
- Hydrazine sulphate, 98%, extra pure, Loba Chemie, india.
- Hydrochloric acid, 35.38%, AR, Lab Tech, United States.
- Hydroxylamine hydrochloride, 98%, extra pure, Loba Chemie, india.
- Methanol, 99.9%, HPLC grade, Romil, United Kingdom.
- Sodium acetate anhydrous, 99%, Loba Chemie, india.

- Sodium hydroxide, 98.0%, S. D. Fine-Chem Limited, India

2.1.2 Instruments:

2.1.2.1. Fourier Transform infra-Red:

IR spectra were recorded on an FT-IR spectrometer using ZnSi cell, tensor 27, Bruker, United States.

2.1.2.2. Nuclear magnetic resonance:

^{1}H NMR spectra were measured on FT-NMR Spectrometer (600 MHz) using TMS as internal standard and $CDCl_3$ as solvent, Bruker, United States.

Or on FT-NMR Spectrometer (300 MHz) using TMS as internal standard and $CDCl_3$ as solvent, Bruker, United States.

2.1.2.3. Gas chromatography mass spectroscopy:

The mass spectra were determined on an electron impact mass spectrometer coupled with gas chromatograph, QP2012, Shimadzu, Japan.

The conditions used for operation below:

Total flow: 50.0 ml/min

Column flow: 1.24 ml/min

Column temperature: 100 ºC

Injection temperature: 290 ºC

Oven temperature program:

Rate	Temperature	Hold time
-	100	0.0
30	160	0.0
50	290	35

Ion source temperature: 200 ºC

Interface temperature: 270 ºC

Start time: 3.00 min

End time: 40 min

2.1.2.4. Ultraviolet-visible spectroscopy:

Ultraviolet-visible spectra were measured using variable wavelengths (200-700 nm) photo diode array connected to liquid chromatograph, using methanol HPLC grade as solvent. The injected sample was passed through union (column free), 2996 Waters, United States.

2.1.2.5. Ultrasound path

The sonication has performed in electronic ultrasonic bath 35 KHz – 80/320 w, RK100, Bandelin, Germany.

2.1.2.6. Microwave oven

Microwave irradiation has performed using microwave oven with 2.45 GHz and 700 w, MS1944W.CWHQDTH, LG, PRC.

2.1.2.7. Thin layer chromatography

TLC was carried out using pre-coated TLC silica gel, aluminium sheets, Merck, Germany, and glass plates using preparative silica gel, Sigma Aldrich, United States.

2.2 General Methods

2.2.1. Synthesis of 1,3- diaryl-prop-2-en-1-ones (I-XV) :

In a 250 mL round bottom flask were placed aromatic aldehyde (0.020 mole), acetophenone or (or its derivatives) (0.025 mole), NaOH (0.01 mole, 0.40 g) dissolved in 5 mL distill water, and 50 mL ethanol. The reaction mixture was sonocated for 1 hour, neutralized with sulfuric acid, 50 ml of cold water was added, and placed in an ice water. The precipitated product was filtered, washed with excess of cold water, dried at ambient temperature and recrystallized from the suitable solvent.

2.2.2. Synthesis of 2-amino- 4,6-diaryl-pyrimidine (XVI-XXIX):

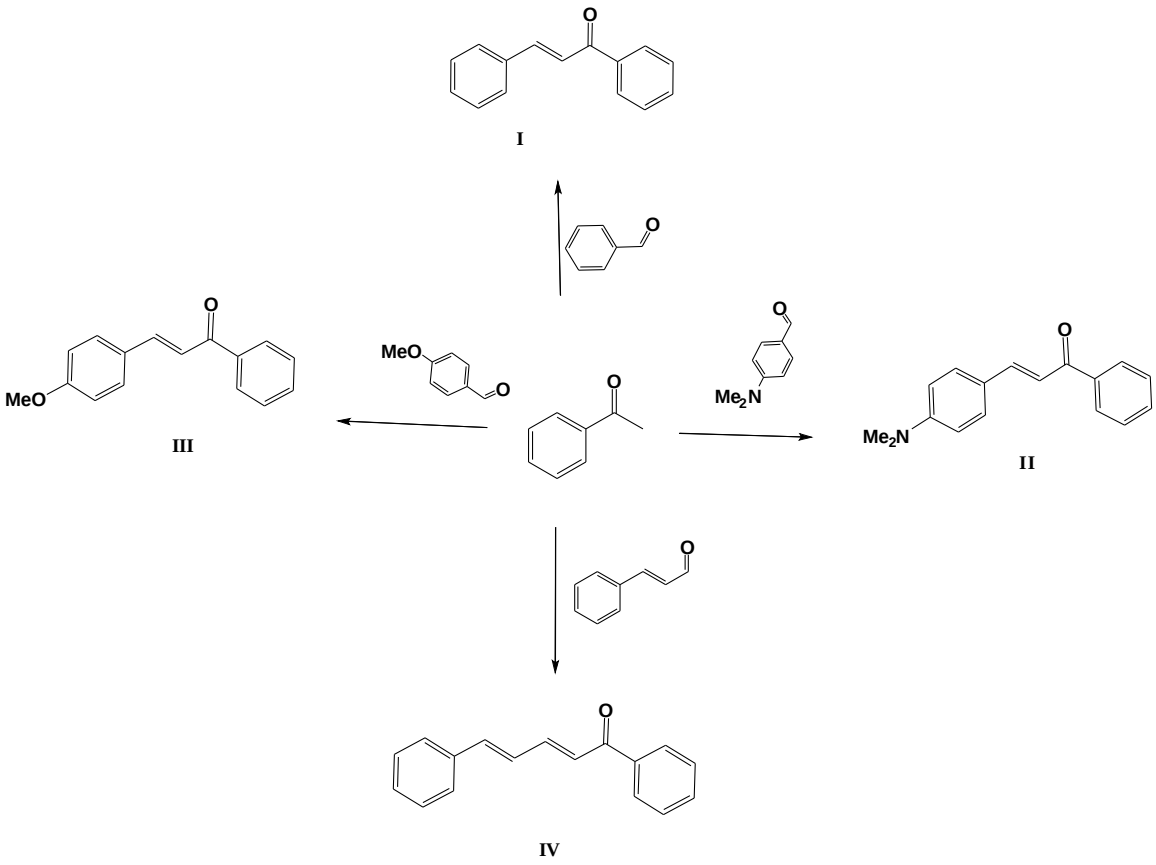
In a 500 ml conical flask were placed the required chalcone (0.001 mole), NaOH (0.003 mole, 0.12 g), guanidine hydrochloride (0.003 mole, 0.29 g) and 100 ml ethanol. The reaction mixture was sonocated for 15 minutes, and transferred to a microwave oven for 30 minutes, the completion of reaction was monitored by TLC. The remaining solvent was removed and the products were purified *via* preparative TLC.

2.2.3. Synthesis of 3,5-diaryl-pyrazoles (XXX-XLIII):

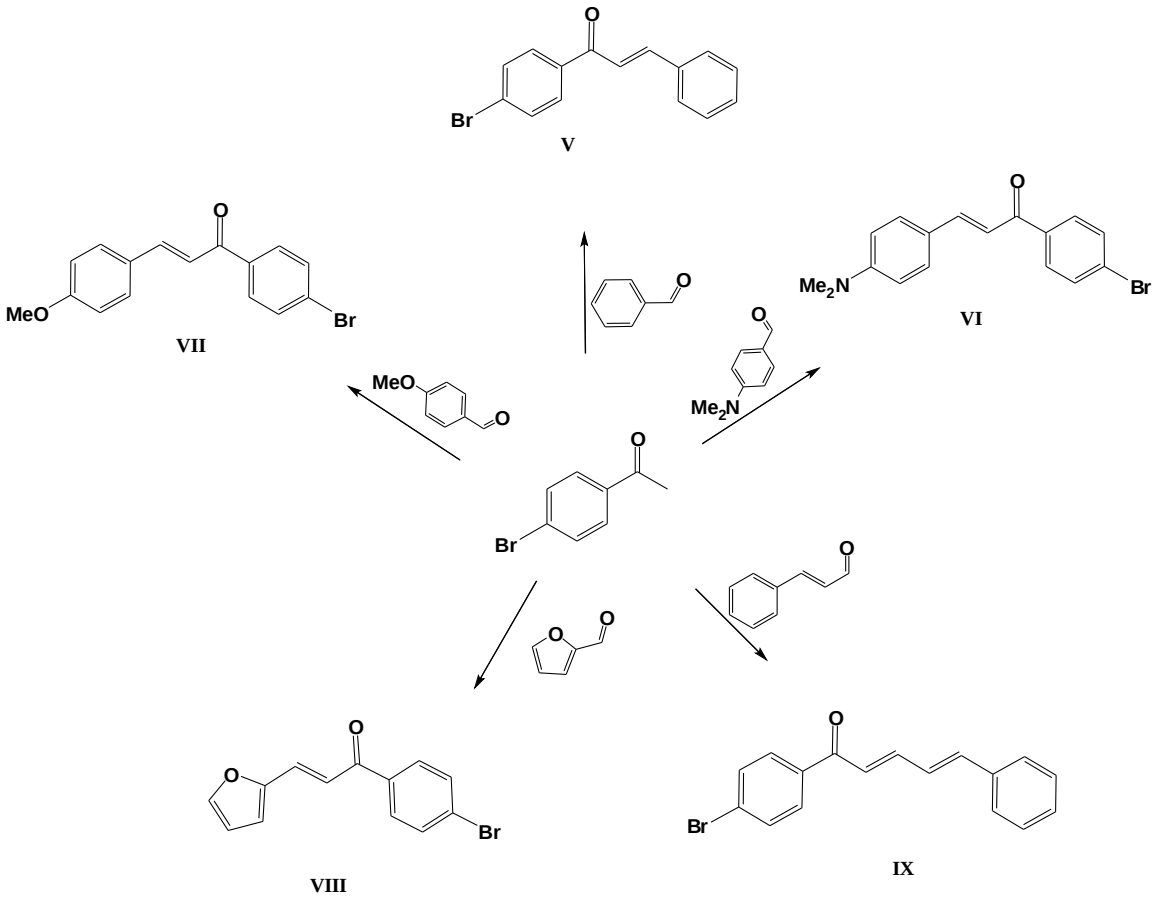
In a 500 ml conical flask were placed the required chalcone (0.001 mole), sodium acetate (0.003 mole, 0.25 g), hydrazine sulfate (0.003 mole, 0.39 g) and 100 ml ethanol. The reaction mixture was sonocated for 15 minutes, and transferred to a microwave oven for 30 minutes, the completion of reaction was monitored by TLC after 10, 20 and 30 minutes. The remaining solvent was removed and the products were purified *via* preparative TLC.

2.2.4 Synthesis of 3,5-diaryl-isoxazoles (XLIV-LVII)

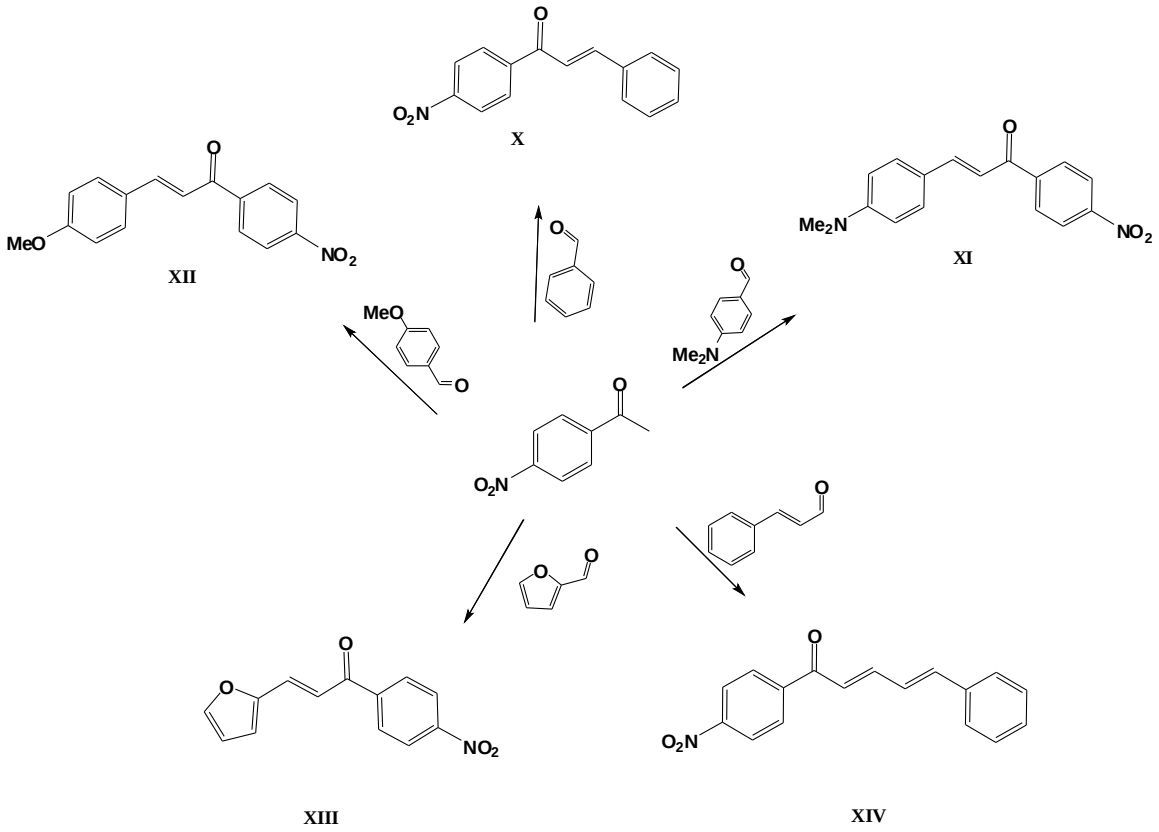
In a 500 ml conical flask were placed the required chalcone (0.001 mole), sodium acetate (0.003 mole, 0.25 g), hydroxylamine hydrochloride (0.003 mole, 0.21 g) and 100 ml ethanol. The reaction mixture was sonocated for 15 minutes, and transferred to a microwave oven for 30 minutes, the completion of reaction was monitored by TLC after 10, 20 and 30 minutes. The remaining solvent was removed and the products were purified *via* preparative TLC.



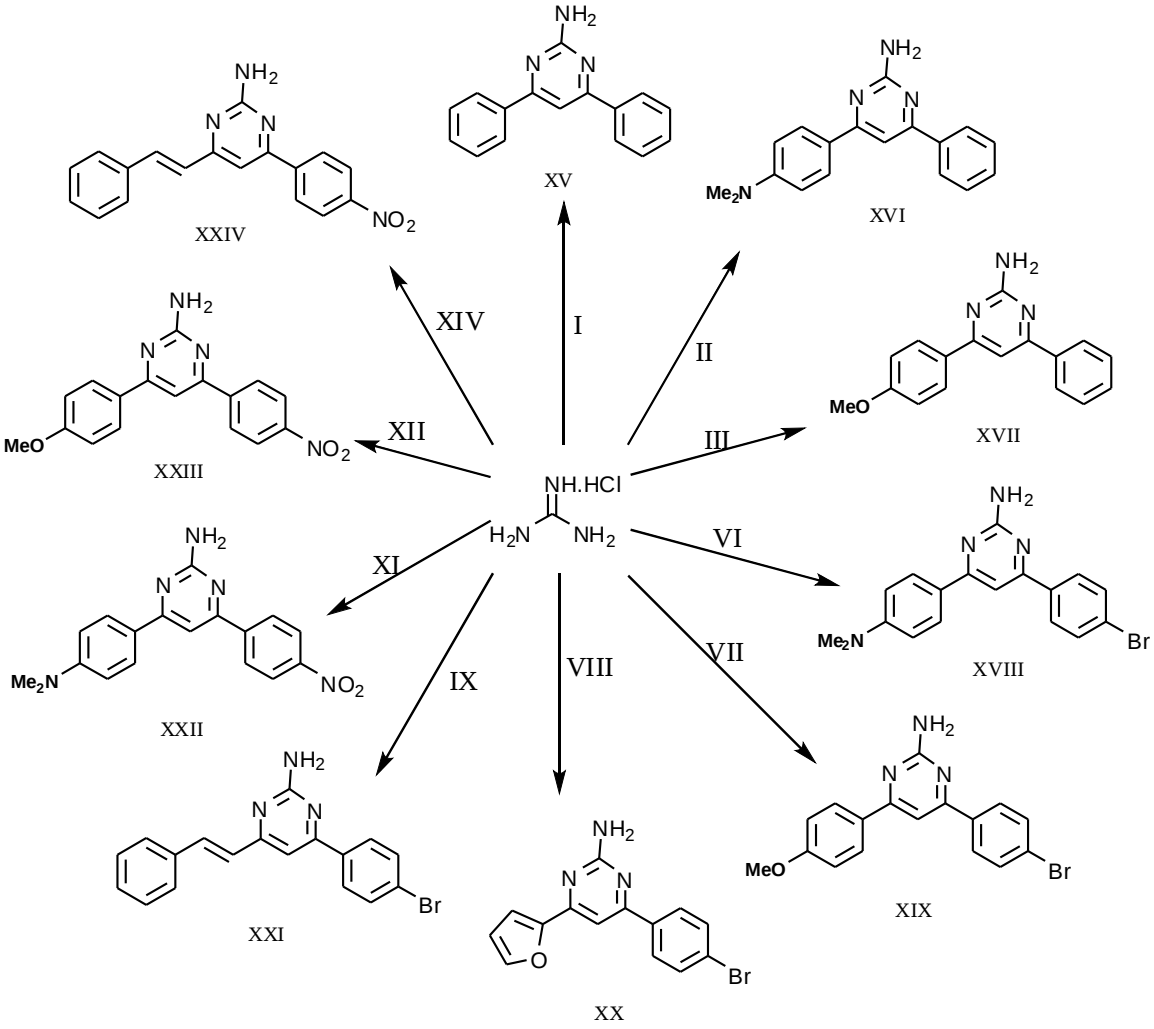
Scheme 2.1: Chemical structure of synthesized 3-aryl-1-phenyl-2propen-1-ones.



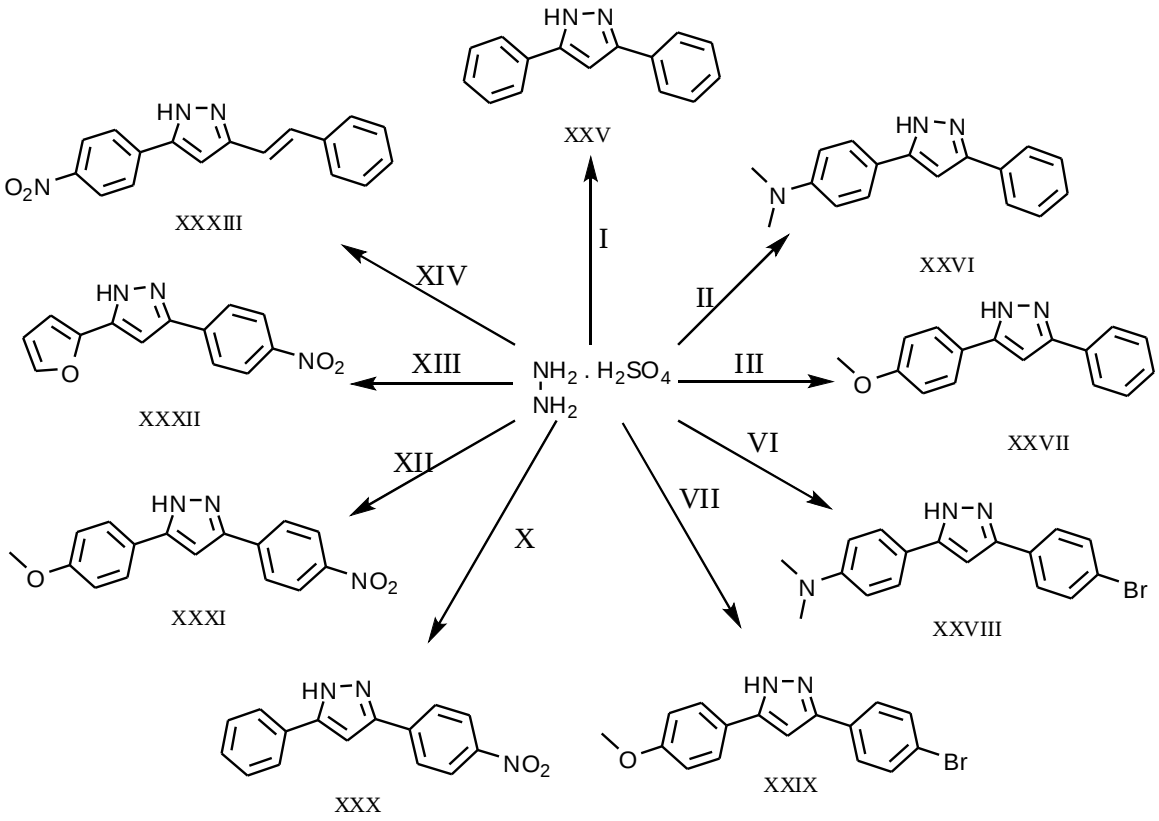
Scheme 2.2: Chemical structure of synthesized 3-aryl-1-(*p*-bromophenyl)-2-propen-1-ones.



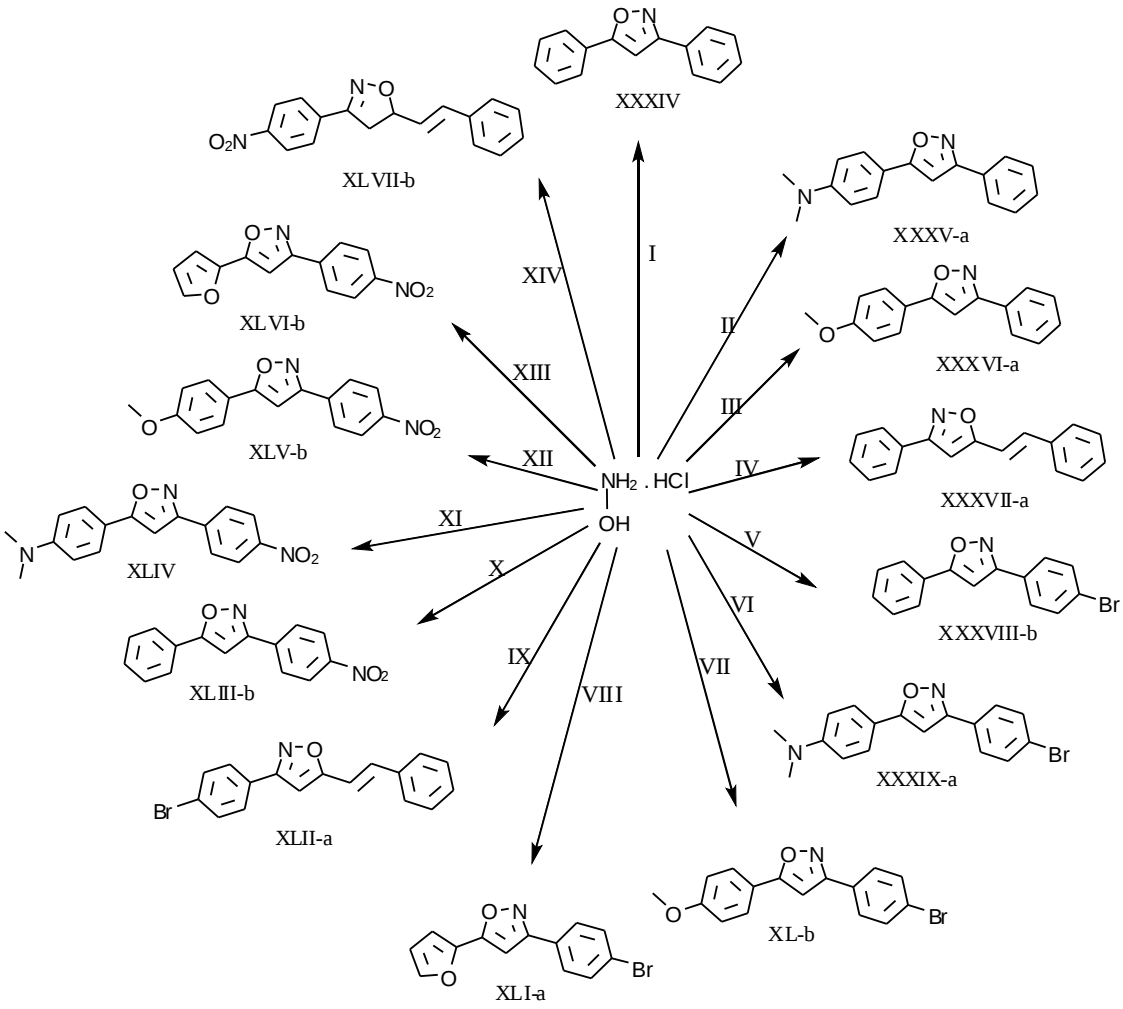
Scheme 2.3: Chemical structure of synthesized 3-aryl-1-(*p*-nitrophenyl)-2-propen-1-ones.



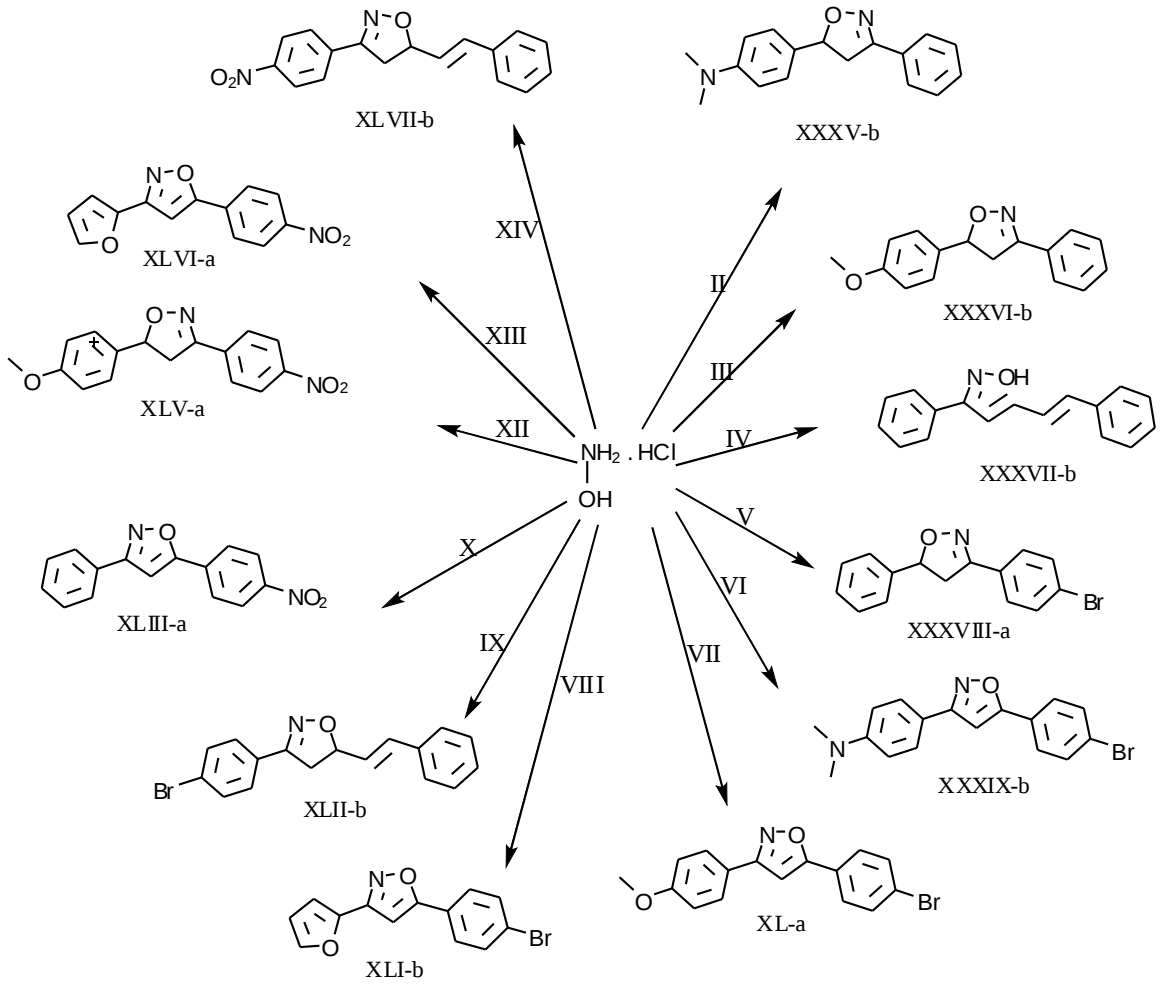
Scheme 2.4: Chemical structure of synthesized 2-amino- 4,6-diaryl-pyrimidine.



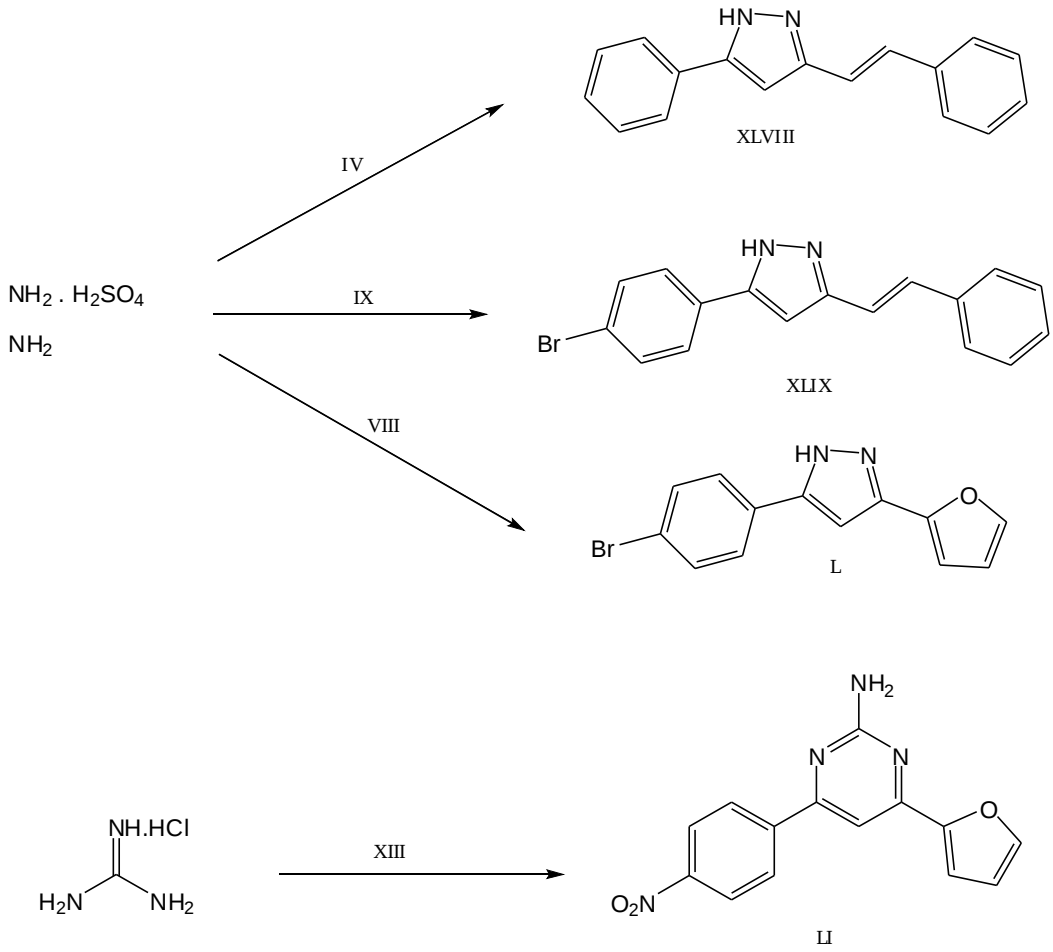
Scheme 2.5: Chemical structure of synthesized 3,5-diaryl-pyrazole.



Scheme 2.6: Chemical structure of synthesized 3,5-diaryl-isoxazole.



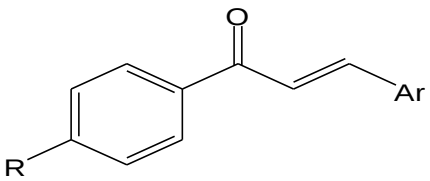
Scheme 2.7: Chemical structure of 4,5 dihydro and regioisomer of 3,5-diaryl-isoxazole.



Scheme 2.9: Non complete synthesis of some pyrazoles and pyrimidine observed by GC_MS.

Tables 2.1: Chemical names of the prepared compounds

Table 2.1.1: Chemical names of the prepared 1,3-diaryl-2propen-1-ones



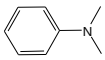
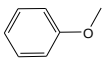
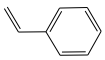
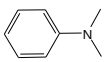
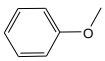
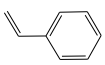
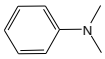
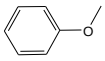
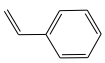
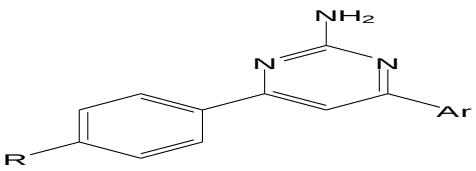
Number	R	Ar	Chemical Name
I	H		1,3-diphenyl-(2E)-propen-1-one
II	H		3-(*p*-N,N dimethylaminophenyl)-1-phenyl-(2E)-propen-1-one
III	H		3-(*p*-methoxyphenyl)-1-phenyl-(2E)-propen-1-one
IV	H		1,5-diphenyl-(2E,4E)-pentadiene-1-one
V	Br		1-(*p*-bromophenyl)-3-phenyl-(2E)-propen-1-one
VI	Br		1-(*p*-bromophenyl)-3-(*p*-N,N dimethylaminophenyl)-(2E)- propen-1-one
VII	Br		1-(*p*-bromophenyl)-3-(*o*-hydroxylphenyl)-(2E)-propen-1-one
VIII	Br		1-(*p*-bromophenyl)-3-(*o*-furyl)-(2E)-propen-1-one
IX	Br		1-(*p*-bromophenyl)-5-phenyl-(2E,4E)-pentadiene-1-one
X	NO_2		1-(*p*-nitrophenyl)-3-phenyl-(2E)-propen-1-one
XI	NO_2		1-(*p*-nitrophenyl)-3-(*p*-N,N dimethylaminophenyl)-(2E)- propen-1-one
XII	NO_2		1-(*p*-nitrophenyl)-3-(*o*-hydroxyllphenyl)- (2E)-propen-1-one
XIII	NO_2		1-(*p*-nitrophenyl)-3-(*o*-furyl)-(2E)-propen-1-one
XIV	NO_2		1-(*p*-nitrophenyl)-5-phenyl-(2E,4E)-pentadiene-1-one

Table 2.1.2: Chemical names of the prepared 2- amino 4,6-diaryl-pyrimidine



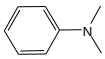
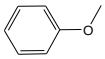
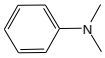
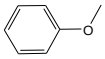
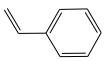
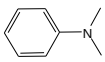
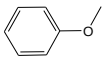
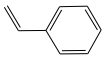
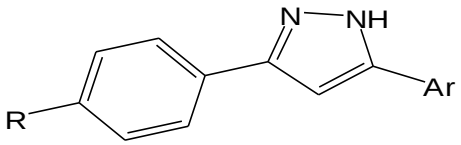
Number	R	Ar	Chemical Name
XV	H		amino 4,6-diphenyl-pyrimidine -2
XVI	H		amino 4-(*p*-N,N dimethylaminophenyl) 6-phenyl pyrimidine-2
XVII	H		amino 4-(*p*-methoxyphenyl) 6-phenyl pyrimidine -2
XVIII	Br		amino 4-(*p*-bromophenyl) 6-(*p*-N,N dimethylaminophenyl) pyrimidine -2
XIX	Br		amino 4-(*p*-bromophenyl) 6-(*p*-methoxyphenyl) pyrimidine -2
XX	Br		amino 4-(*p*-bromophenyl) 6-(furyl) pyrimidine -2
XXI	Br		amino 4-(*p*-bromophenyl) 6-(ethenyl-2-phenyl) pyrimidine -2
XXII	NO_2		amino 4-(*p*-nitrophenyl)-6-(*p*-N,Ndimethylaminophenyl)-pyrimidine -2
XXIII	NO_2		amino 4-(*p*-nitrophenyl)-6-(*p*-methoxyphenyl) pyrimidine -2
XXIV	NO_2		amino 4-(*p*-nitrophenyl)-6-(ethenyl-2-phenyl) pyrimidine -2

Table 2.1.3: Chemical names of the prepared 3,5-diphenyl-pyrazole



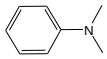
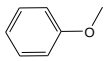
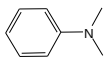
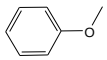
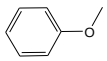
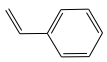
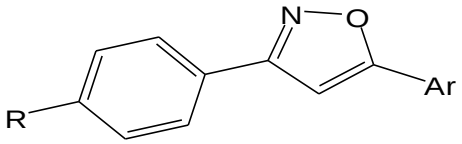
Number	R	Ar	Chemical Name
XXV	H		3,5-diphenyl-pyrazole
XXVI	H		3-phenyl-5-(*p*-N,Ndimethylaminophenyl)-pyrazole
XXVII	H		3-phenyl-5-(*p*-methoxyphenyl)- pyrazole
XXVIII	Br		3-(*p*-bromophenyl)-5-(*p*-N,Ndimethylaminophenyl)-pyrazole
XXIX	Br		3-(*p*-bromophenyl)-5-(*p*-methoxyphenyl)-pyrazole
XXX	NO_2		3-(*p*-nitrophenyl)-5-phenyl-pyrazole
XXXI	NO_2		3-(*p*-nitrophenyl)-5-(*p*-methoxyphenyl)-pyrazole
XXXII	NO_2		3-(*p*-nitrophenyl)-5-(furyl)-pyrazole
XXXIII	NO_2		3-(*p*-nitrophenyl)-5-(ethenyl-2-phenyl)-pyrazole

Table 2.1.4: Chemical names of the prepared and purified 3,5-diphenyl-isoxazole



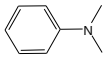
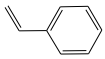
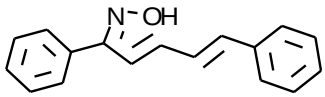
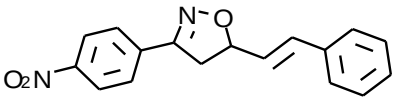
Number	R	Ar	Chemical Name
XXXIV	H		diphenyl-isoxazole-3,5
XLIV	NO_2		3-(*p*-nitrophenyl)-5-(4-N,Ndimethylaminophenyl)-isoxazole
XLVII-a	NO_2		3-(*p*-nitrophenyl)-5-(ethenyl-2-phenyl)-isoxazole

Table 2.1.5: Chemical names of the prepared and isolated isoxazole intermediate



XXXVII-b

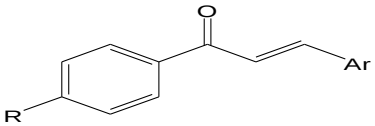


XLVII-b

Number	Chemical Name
XXXVII-b	1,5- diphenylpenta- (2E,4E)-dien-1 oxime
XLVII-b	3-(*p*-nitrophenyl)-5-(ethenyl-2-phenyl)-4,6-dihydroisoxazole

Tables 2.2 Reaction conditions of the prepared compounds

Table 2.2.1: Reaction conditions of the prepared 1,3-diaryl-2E-propen-1-ones



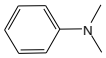
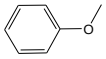
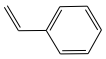
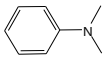
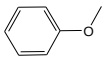
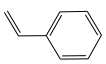
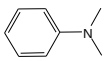
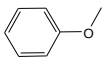
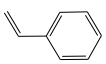
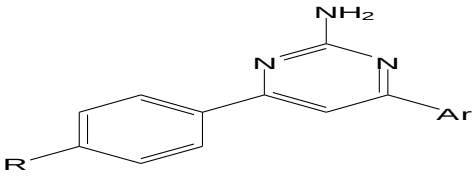
Number	R	Ar	Solvent	Reaction temperature	Yield %/(g)	Recryst-allization solvent	Mating point
I	H		EtOH	Room temp. (27-33 Cº)	55/2.29	MeOH	55-57 (Mahajan et al, 2010)
II	H		EtOH	Room temp. (27-33 Cº)	74/3.71	EtOH	107-108 (CSID: 4526350)
III	H		EtOH	Room temp. (27-33 Cº)	66/3.14	EtOH	76-78 (Silva et al, 2010)
IV	H		EtOH	Room temp. (27-33 Cº)	72/3.37	EtOH	84-86 (Wendelin et al, 1985)
V	Br		EtOH	Room temp. (27-33 Cº)	20/1.15	Acetone-H_2O	91-92 (Kumar et al, 2011)
VI	Br		EtOH	Room temp. (27-33 Cº)	99/6.53	Acetone-H_2O	142-143 (Guoxi et al, 2011)
VII	Br		EtOH	Room temp. (27-33 Cº)	90/5.71	Acetone-H_2O	147-149 (CSID: 4512945)
VIII	Br		EtOH	Room temp. (27-33 Cº)	91/5.04	Acetone-H_2O	66-68 (CSID: 4687757)
IX	Br		EtOH	Room temp. (27-33 Cº)	96/6.01	Acetone-H_2O	153-154 (CSID: 1267188)
X	NO_2		EtOH	Room temp. (27-33 Cº)	41/2.07	Acetone-H_2O	94-95 (Cocconcelli et al, 2008)
XI	NO_2		EtOH	Room temp. (27-33 Cº)	64/3.79	Acetone-H_2O	207-208 (Kumar 2006)
XII	NO_2		EtOH	Room temp. (27-33 Cº)	74/4.19	Acetone-H_2O	166-168 (Kumar 2006)
XIII	NO_2		EtOH	Room temp. (27-33 Cº)	98/4.76	Acetone-H_2O	119-121 (CSID: 4650601)
XIV	NO_2		EtOH	Room temp. (27-33 Cº)	95/5.3	Acetone-H_2O	173-174 (CSID: 4800250)

Table 2.2.2: Reaction conditions of the prepared 2- amino 4,6-diaryl-pyrimidine



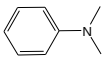
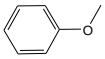
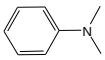
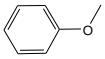
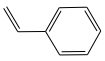
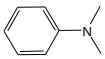
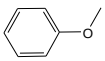
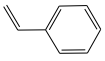
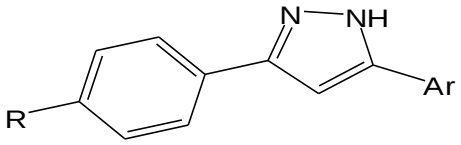
Number	R	Ar	Solvent	Reaction temperature	Yield %/ (g)	Color	Mating point
XV	H		EtOH	Room temp. (27-33 C°)	88/0.22	Yellow	192-194 (Varga et al, 2003)
XVI	H		EtOH	Room temp. (27-33 C°)	64/0.19	Dark-Yellow	152-153 (Thanh et al, 2009)
XVII	H		EtOH	Room temp. (27-33 C°)	79/0.22	Dark-Yellow	150-151 (213, Kumar et al, 2011)
XVIII	Br		EtOH	Room temp. (27-33 C°)	50/0.18	Light-Brown	Above 250 (Thanh et al, 2009)
XIX	Br		EtOH	Room temp. (27-33 C°)	50/0.18	Light-Brown	119-120 (Thanh et al, 2009)
XX	Br		EtOH	Room temp. (27-33 C°)	51/0.16	Brown	174-175 (Joshi et al 2012)
XXI	Br		EtOH	Room temp. (27-33 C°)	37/0.13	Brown	Above 250 (Wendelin et al 1985)
XXII	NO_2		EtOH	Room temp. (27-33 C°)	46/0.15	Brown	187-188 (Kumar 2006)
XXIII	NO_2		EtOH	Room temp. (27-33 C°)	43/0.14	Brown	153-154 (191, Kumar et al, 2011)
XXIV	NO_2		EtOH	Room temp. (27-33 C°)	46/0.15	Dark-Yellow	177-179 (Wendelin et al, 1985)

Table 2.2.3: Reaction conditions of the prepared 3,5-diphenyl-pyrazole



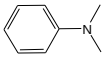
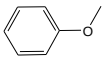
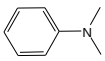
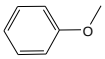
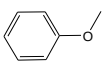
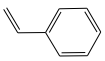
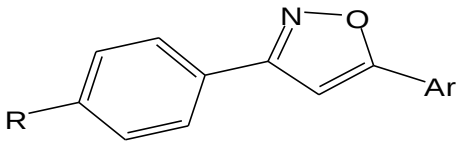
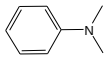
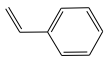
Number	R	Ar	Solvent	Reaction temperature	Yield %/ (g)	Color	Mating point
XXV	H		EtOH	Room temp. (27-33 Cº)	97/0.21	Dark-Yellow	186-188 (Singh et al, 2011)
XXVI	H		EtOH	Room temp. (27-33 Cº)	35/0.09	Brown	227-229 (CSID: 5044606)
XXVII	H		EtOH	Room temp. (27-33 Cº)	94/0.24	Yellow	153-154 (158, Fullam et al, 2013)
XXVIII	Br		EtOH	Room temp. (27-33 Cº)	23/0.08	Brown	204-206
XXIX	Br		EtOH	Room temp. (27-33 Cº)	61/0.20	Dark-Yellow	189-191 (Shaw et al, 2010)
XXX	NO_2		EtOH	Room temp. (27-33 Cº)	23/0.06	Dark-Yellow	236-238 (199, Shaw et al, 2010)
XXXI	NO_2		EtOH	Room temp. (27-33 Cº)	26/0.08	Brown	193-194 (Shaw et al, 2010)
XXXII	NO_2		EtOH	Room temp. (27-33 Cº)	33/0.08	Brown	218-220 (CSID: 3494923)
XXXIII	NO_2		EtOH	Room temp. (27-33 Cº)	37/0.11	Brown	216-218 (CID 6611215)

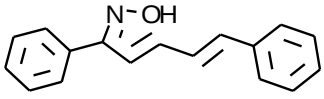
Table 2.2.4: Reaction conditions of the prepared and purified 3,5-diphenyl-isoxazole



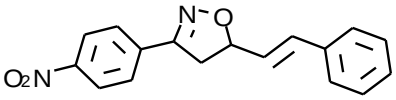
Number	R	Ar	Solvent	Reaction temperature	Yield % / (g)	Color	Mating point
XXXIV	H		EtOH	Room temp. (27-33 C°)	91/ 0.20	Yellow	154-155 (Ueda et al, 2011)
XLIV	NO_2		EtOH	Room temp. (27-33 C°)	33/ 0.10	brown	132-133
XLVII-a	NO_2		EtOH	Room temp. (27-33 C°)	Low yield	Yellow-green	182-184

* Isolated product

Table 2.2.5: Reaction conditions of the prepared and isolated isoxazole intermediate



XXXVII-b

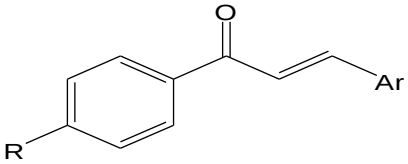


XLVII-b

Number	Solvent	Reaction temperature	Yield %	Color
XXXVII-b	EtOH	Room temp. (27-33 C°)	Minor product	White
XLVII-b	EtOH	Room temp. (27-33 C°)	Minor product	brown

Tables 2.3: Infrared spectral data of the prepared compounds

Table 2.3.1: Infrared spectral data of the prepared 1-aryl-3-phenyl-prop-2-en-1-ones



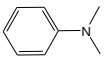
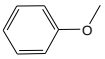
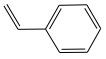
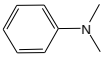
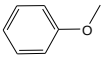
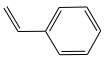
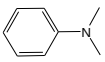
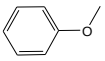
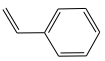
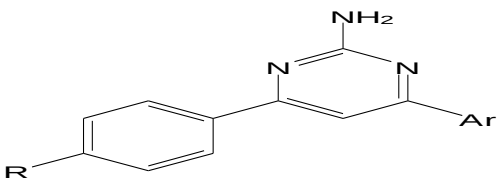
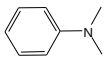
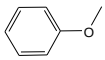
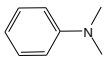
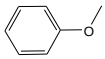
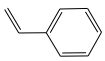
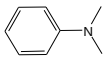
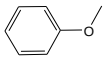
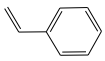
Number	R	Ar	C=O St-vib	C=C St-vib	C-H St-vib	C-O St-vib	$-NO_2$ St-vib	C-H Bn-vib (out of plan)
I	H		1661	1575, 1603	3061	-	-	mono 687, 744 para
II	H		1647	1579, 1596	3040	-	-	mono 689, 777 para 814
III	H		1656	1577, 1588	3065	1257	-	mono 688, 779 para 824
IV	H		1653	1576, 1586, 1599	3062	-	-	mono 690, 752
V	Br		1673	1583, 1602	2967	-	-	mono 692, 761 para 827
VI	Br		1663	1565, 1582	2960	-	-	para 809
VII	Br		1656	1565, 1592	2950	1256	-	para 811
VIII	Br		1678	1584, 1655	3078	-	-	para 815
IX	Br		1648	1567, 1581, 1597	3027	-	-	mono 684, 746 para 812
X	NO_2		1662	1573, 1590, 1603	2961	-	1514, 1334	mono 680, 739 para 844
XI	NO_2		1646	1565, 1581	3101	-	1515, 1338	para 804, 855
XII	NO_2		1656	1572, 1584, 1603	3113	1257	1512, 1339	para 802, 855
XIII	NO_2		1657	1548,1587	3145	-	1522, 1343	para 818
XIV	NO_2		1654	1570, 1598	3112	-	1521, 1345	mono 691, 753 para 827

Table 2 mono= monosubstituted ring
para= paradi substituted ring

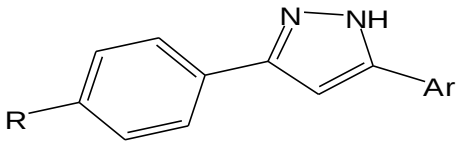
red 2- amino 4,6-diaryl-pyrimidine



Num-ber	R	Ar	St-vib	St-vib	St-vib	St-vib	St-vib	St-vib	Bn-vib (out of plan)	Bn-vib (out of plan)
XV	H		1586, 1604, 1623	1544, 1566	2923	-	3189, 3312	-	Overlapped with 760 b	mono 690, 760 b
XVI	H		1584, 1606 b	1496, 1527, 1561	2923	-	3189, 3311	-	767	mono 694, 754 para 817
XVII	H		1568, 1608, 1643	1497, 1514, 1536, 1562	2936	1176	3192, 3324	-	770	mono 686, 754 para 821
XVIII	Br		1585, 1606	1501, 1514, 1536, 1567	2935	-	3197, 3317	-	771	para 828
XIX	Br		1578, 1607	1488, 1512, 1533, 1563	2924	1178	3183, 3351	-	772	para 816
XX	Br		1576, 1600	1488, 1509, 1535, 1556	2920	-	3188, 3327	-	772	para 815
XXI	Br		1577, 1589, 1650	1462, 1494, 1529, 1561	2923	-	3199, 3332	-	Overlapped with 772 b	mono 699, 772 b para 810
XXII	NO_2		1605	1494, 1565	2982	-	3197, 3334	1536, 1348	772	para 815
XXIII	NO_2		1604	1514, 1568	2931	1177	3197, 3324	1540, 1348	760	para 825
XXIV	NO_2		1577, 1589, 1651	1487, 1561	2922	-	3190, 3327	1530, 1362	772	mono 693, 755 para 810

mono= monosubstituted ring
para= paradi substituted ring

Table 2.3.3: Infrared spectral data of the prepared 3,5-diphenyl-pyrazole



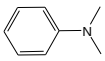
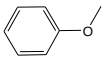
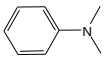
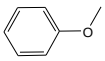
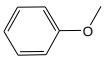
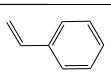
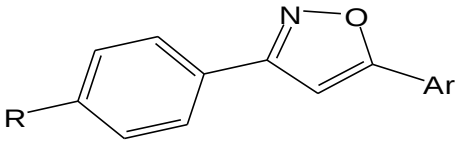
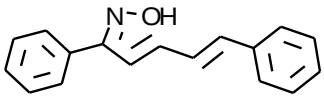
Number	R	Ar	St-vib	St-vib	St-vib	St-vib	St-vib	Bn-vib (out of plan)	Bn-vib (out of plan)
XXV	H		1462, 1495	3004	3134	-	-	771	mono 687, 753 para 838
XXVI	H		1461, 1524, 1616	2924	3167	-	-	772	mono 702 para 820
XXVII	H		1460, 1508, 1614	2924	3129	-	1252	771	mono 691, para 833
XXVIII	Br		1443, 1522, 1617	2922	3219	-	-	772	para 818
XXIX	Br		1438, 1512, 1615	2923	3229	-	1250	772	para 830
XXX	NO_2		,1458 ,1497 1602	2923	3184	1519 ,1334	-	772	mono 685, para 853
XXXI	NO_2		1454, 1602, 1616	2923	3134	1518 ,1340	1254	773	mono 834, para 854
XXXII	NO_2		,1474 1602	2923	3240	1509 ,1342	-	773	para 853
XXXIII	NO_2		,1448 1602	2923	3155	1516 ,1339	-	772	para 854

Table 2.3.4: Infrared spectral data of the prepared and purified 3,5-diphenyl-isoxazole

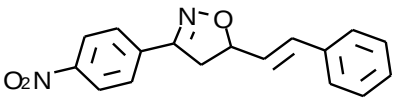


Number	R	Ar	St-vib	St-vib	St-vib	$-NO_2$	Bn-vib (out of plan)	Bn-vib (out of plan)
XXXIV	H		1457	2924	1511, 1604	-	773	mono 700, 773
XLIV	NO_2		1475	2922	1574, 1612	1342, 1520	772	para 835
XLVII-a	NO_2		1441	2922	1578, 1604	1347, 1521	779	mono 706 para 855

Table 2.3.5: Infrared spectral data of the prepared and isolated isoxazole intermediates



XXXVII-b

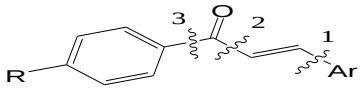


XLVII-b

Number	St-vib	St-vib	St-vib	$-NO_2$	O-H	C=C	Bn-vib (out of plan)	Bn-vib (out of plan)
XXXVII-b	2923	1495, 1447	-	-	3268	1685, 1602	-	mono 693, 750
XLVII-b	2923	1518, 1493	1450	1345, 1576	-		772	momo 691, 751 para 850

Tables 2.4 Mass spectral data of the prepared compounds

Table 2.4.1: Mass spectral data of the prepared 1-aryl-3-phenyl-prop-2-en-1-ones



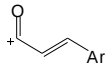
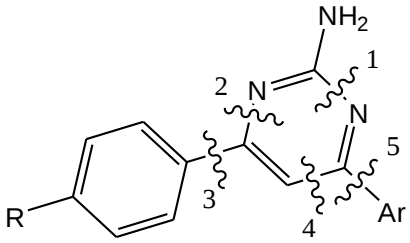
Num-ber	R	Ar	Calculated MW	Observed $M^{+\cdot}$ (M/Z)	2: (M/Z)	3a:  (M/Z)	3b:  (M/Z)	1: Ar^{+} (M/Z)
I	H		208.09	208	103	77	131	77
II	H		251.13	251	146	77	174	121
III	H		238.10	238	133	77	161	108
IV	H		234.10	234	129	77	157	103
V	Br		287.15	285-287	103	155,157	131	77
VI	Br		330.22	329-331	146	155,157	174	121
VII	Br		317.18	316-318	133	155,157	161	108
VIII	Br		277.11	276-278	93	155,157	121	-
IX	Br		313.19	312-314	129	155,157	157	103
X	NO_2		253.25	253	103	-	131	77
XI	NO_2		296.32	296	146	-	174	121
XII	NO_2		283.28	283	133	-	161	108
XIII	NO_2		243.21	243	93	-	121	-
XIV	NO_2		279.29	279	129	-	157	103

Table 2.4.2: Mass spectral data of the prepared 2- amino 4,6-diaryl-pyrimidine



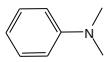
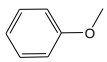
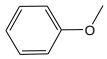
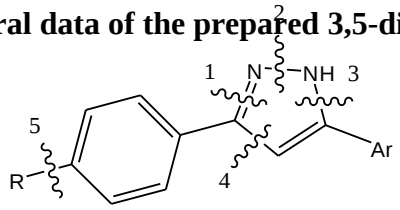
Number	R	Ar	Calculated MW	Observed $M^{+\cdot}$ (M/Z)	1+2: (M/Z)	3+5:  (M/Z)	4:  (M/Z)	Phenyl^{+} (M/Z)	4+1:  (M/Z)
XV	H		247.29	247	204	51	102	77	102
XVI	H		290.36	290	247	51	102	77	145
XVII	H		277.32	277	234	51	102	77	132
XIX	Br		356.22	355, 357	313, 315	51	102	77	132
XX	Br		316.15	315, 317	272, 274	51	102	77	92
LI	NO_2		282	282	-	51	102	77	92

Table 2.4.3: Mass spectral data of the prepared 3,5-diphenyl-pyrazole



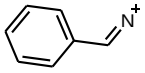
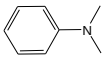
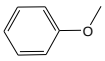
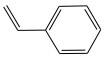
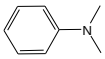
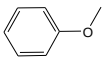
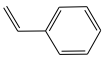
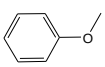
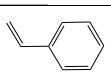
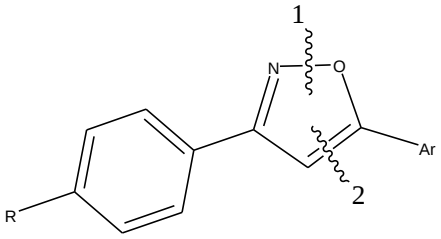
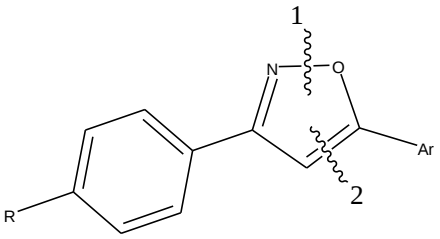
Number	R	Ar	Calculated MW	Observed $M^{+\cdot}$ (M/Z)	1+3: M-N_2 (M/Z)	Phenyl$^+$ (M/Z)	2+4+5:  (M/Z)
XXV	H		220.27	220	191	77	104
XXVI	H		263.34	263	234	77	104
XXVII	H		250.30	250	221	77	104
XLVIII	H		246	264	218	77	104
XLIX	Br		299	298,300	272	77	104
XXVIII	Br		342.23	343 ,341	-	77	104
XXIX	Br		329.19	330 ,328	-	77	104
XLIX	Br		325	328 ,326	-	77	104
L	Br		289	290 ,288	262	77	104
XXX	NO_2		265.27	265	235	77	104
XXXI	NO_2		295.29	295	265	77	104
XXXII	NO_2		255.23	255	225	77	104
XXXIII	NO_2		291.30	291	261	77	104

Table 2.4.4: Mass spectral data of the prepared 3,5-diphenyl-isoxazole



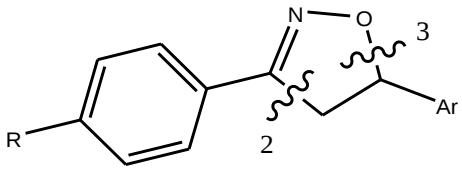
Number	R	Ar	Calculated MW	Observed $M^{+\cdot}$ (M/Z)	1+2  (M/Z)	4: M-Phenyl^{+} (M/Z)
XXXIV	H		221.25	221	105	144
XXXV-a	H		264.32	264	148	-
XXXVI-a	H		251.28	251	135	174
XXXVII-a	H		247.29	247	131	-
XXXVIII-a	Br		300.15	299,301	105	-
XXXIX-a	Br		343.22	342,344	148	159
XL-a	Br		330.18	329,331	135	-
XLI-a	Br		290.11	289,291	95	-
XLII-a	Br		326.19	325,327	131	-
XLIII-a	NO_2		266.25	266	105	-
XLIV	NO_2		309.32	309	148	-
XLV	NO_2		296.28	296	135	-
XLVI-a	NO_2		256.21	265	95	
XLVII-a	NO_2		292.29	292	131	215

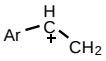
Table 2.4.5: Mass spectral data of the prepared isoxazole regioisomers

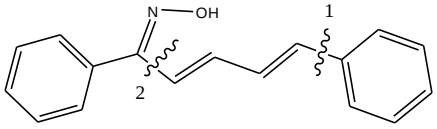


Number	**R**	**Ar**	**Calculated MW**	**Observed $M^{+\cdot}$ (M/Z)**	**1+2**  **(M/Z)**	**Phenyl^{+} (M/Z)**
XXXIX-a	Br		243.22	342,344	-	77
XL-a	Br		330.18	329,331	135	77
XLI-a	Br		290.11	289,291	-	77
XLIII-b	NO_2		266.25	266	-	
XLVI-b	NO_2		256.21	265	95	

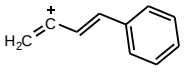
Table 2.4.6: Mass spectral data of the prepared isoxazole intermediates



Number	R	Ar	Calculated MW	Observed $M^{+\cdot}$ (M/Z)	1+2  (M/Z)	4:  (M/Z)	Phenyl^{+} (M/Z)
XXXV-b	H		266.34	266	-	147	77
XXXVI-b	H		253.30	253	-	134	77
XXXVIII-a	Br		302.17	301,303	-	104	77
XLII-a	Br		328.20	327,329	-	129	77
XLV-a	NO_2		298.29	298	-	134	77
XLVII-a	NO_2		294.30	294	-	129	77



XXXVII-b

Number	Calculated MW	Observed $M^{+\cdot}$ (M/Z)	2 or 2+3:  (M/Z)	1: M-Phenyl^{+} (M/Z)	1: Phenyl^{+} (M/Z)
XXXVII-b	249.31	249	129	172	77